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Applicant FISHER, Tim et al	

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A1

DEMANDE DE BREVET D'INVENTION

(21) **N° 75 21073**

(54) **Procédé pour l'obtention d'aliments protéiques à partir de tourteaux ou graines d'origine végétale.**

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La présent inv ntion concerne un procédé pour l'obt ntion d'aliments protéiqu s à partir de tourteaux ou graines d'origine végétale; elle a également pour objet des aliments protéiques ainsi obtenus, ceux-ci possédant des caractéristiques organoleptiques semblables à celles de la viande.

Il est connu qu'actuellement on cherche de nouvelles sources de protéines pour répondre aux besoins de l'alimentation aussi bien des pays industrialisés que des pays en voie de développement.

Parmi les sources de protéines utilisées de nos jours, on peut citer notamment les tourteaux et graines d'origine végétale, par exemple les graines de soja, d'arachide, de tournesol, de féverole, de colza et les tourteaux issus de ces graines ainsi que les pois et les lupins.

On connaît déjà des procédés pour l'obtention de produits protéiques utilisables pour l'alimentation. On peut notamment se référer aux nombreux procédés connus pour le traitement du soja en vue de l'obtention de protéines concentrées, de protéines isolées, de protéines texturées ou de mélanges lipide-protéines d soja modifiées par un traitement enzymatique. A cet effet on peut se référer par exemple aux travaux de EDW N.W. ; MEYER (Oilseed protein concentrated and Isolates - J. of AM. oil chemist's soci - ty. 1971 - 48, N° 9, 484 - 488); de RADCLIFFE F. ROBINSON (What is the future of textured protein products ? - Food technology. 1971 - Vol 26 - N° 5, pp 59-63) et de SHEMER M., WEIL. S. and PERKINS E.G. (Nutritional and chemical studies of three processed soybean food - J. of Food Science. 1973 - 38, 112- 115).

L'utilisation du soja dans l'alimentation humaine se heurte cependant à certaines difficultés dont on rappellera ci-après les principales : les graines et la farine de soja ont un goût et une odeur peu agréables; il peut se produire une fermentation dans l'intestin lors d'une forte consommation de produits à base de soja, du fait des galactosides dérivés du saccharose contenus dans le soja (voir à cet effet RACKIS J.J. Biological and Physiological factors in soybeans J. AM. Oil chemist's society 1974 - 51).Il existe des facteurs antinutritionnels dans les graines, la farine crue de soja ou certains produits protéiques en résultant ; ces facteurs antinutritionnels sont notamment l'anti-

trypsine, l'hémagglutinine, l'acide phytique, les saponines, etc....

Des études enzymologiques et technologiques approfondies ont déjà été entreprises pour le traitement de protéines de soja dans le but de surmonter les difficultés ci-dessus et de contribuer à l'amélioration de l'alimentation humaine.

On a maintenant trouvé un nouveau procédé pour l'obtention de produits protéiques à partir de tourteaux ou graines d'origine végétale, lesdits produits protéiques présentant des caractéristiques organoleptiques semblables à celle de la viande et présentant un taux de facteurs anti-nutritionnels inférieur à celui obtenu avec les autres procédés.

Le procédé selon la présente invention consiste :

1) à faire coaguler les protéines à partir d'un produit résultant d'un traitement préalable des graines ou des tourteaux d'origine végétale, ladite coagulation étant réalisée à l'aide d'un sel alcalino-terreux;

2) à traiter sous pression la fraction protéique résultante pour obtenir un fromage de protéines;

3) à porter le fromage de protéines à une température de congélation au plus égale à -5°C; et

4) à décongeler ledit fromage de protéines à une température au moins égale à la température ambiante.

Le produit de départ mis en oeuvre dans le procédé de l'invention est un produit résultant d'un traitement préalable des graines ou des tourteaux d'origine végétale, ledit traitement préalable étant essentiellement constitué d'une étape de trempage dans l'eau et d'une étape de chauffage.

L'étape de trempage est avantageusement réalisée à une température comprise entre 10 et 60°C pendant environ 3 à 8 heures.

Le procédé selon l'invention est applicable aussi bien aux graines entières non-délipidées qu'aux tourteaux d'origine végétale.A titre d'exemples de graines utilisables aux fins de l'invention,on peut citer les graines de soja, d'arachide, de tournesol, de féverole, de colza; on peut également utiliser les tourteaux issus de ces graines ainsi que les pois et les lupins.

Dans le cas où l'on utilise un tourteau d'origine végétale, on réalise avantageusement l'étape de trempage en mélangeant

le tourt au avec 8 à 10 fois son volum d'eau, n ajustant le pH à nviron 8, par xempl à l'aide d'hydroxyde d sodium, et en chauffant le mélange résultant à une t mpérature d'environ 40 à 60°C, d préfér nce d'environ 50°C, p ndant environ 3 h ures. Il est particulièrement avantageux d'opérer en présence d'un agent favorisant l'élimination des sucres et des mauvaises odeurs; un tel agent est par exemple le carbonate de sodium (Na_2CO_3). On élimine ensuite du produit résultant les résidus insolubles, par exempl par centrifugation ou filtration; la solution obtenue est alors chauffée à une température comprise entre environ 100 et 120°C, de préférence à 110°C, pendant environ 10 à 20 minutes; ce chauffage favorise la destruction des anti-trypsines. Il est avantageux selon une variante du procédé de l'invention de réaliser l'étape de chauffage en présence d'un agent anti-oxydant. On peut utiliser par exemple le sulfate acide de sodium ($NaHSO_4$) à raison d'environ 150 ppm. On peut en outre rajouter du $Ca(OH)_2$ afin d'activer la destruction du facteur antitrypsique et augmenter le rendement d'extraction. Le produit obtenu après la mise en oeuvre des étapes de trempage et de chauffage constitue ce que l'on appelle dans la présente description "le produit résultant d'un traitement préalable de tourteaux d'origine vététale".

Lorsqu'on utilise des graines d'origine végétale, le traitement préalable est également constitué essentiellement d'une étape de trempage et d'une étape de chauffage. On fait tremper dans l'eau les graines, en présence d'un agent favorisant l'élimination des sucres et des mauvaises odeurs, tels que Na_2CO_3, à une température comprise entre 10 et 20°C environ, de préférence à 15°C; ce trempage dure environ 8 heures; on élimine ensuite l'eau du milieu réactionnel et on rince à l'eau, puis on broye les grains gonflés et on traite à l'aide d'un agent anti-oxydant, tel que $NaHSO_4$ ou $Ca(OH)_2$. On chauffe ensuite le milieu réactionnel à une température comprise entre 100 et 120°C environ, de préférence à 110°C, pendant environ 10 à 20 minutes. On élimine ensuite par filtration les résidus insolubles; la solution résultante constitue ce que l'on appelle dans la présente description " le produit résultant d'un traitement préalable de graines d'origine végétale".

Selon le procédé de l'invention, on fait coaguler les protéines contenues dans le produit résultant d'un traitement préalable de graines ou de tourteaux d'origine végétale, à l'aide

d'un sel alcalino-terr ux à une températur par exemple de 70°C. A titre de sels alcalino-terreux utilisables selon la présent invention,on peut cit r par exemple les chlorures et sulfates de magnésium ou de calcium. On utilise en général le sel alcalino-terreux à raison d'environ 1 à 5% en poids par rapport à l'extrait sec du produit traité. Le caillé de protéines obtenu ou fraction protéique présente des propriétés fonctionnelles intéressantes; ce produit est utilisable en alimentation humaine et/ou comme adjuvant dans l'industrie alimentaire; il possède des propriétés sensiblement identiques à celles obtenues par le procédé connu de précipitation des protéines par l'acide chlorhydrique.

On peut noter par exemple que la viscosité et la solubilité de la fraction protéique selon l'invention ne varient pratiquement pas en fonction de la température.

Par ailleurs,la solubilité d'une telle fraction varie en fonction du pH ; elle est pratiquement insoluble aux alentours de pH 5.

La fraction protéique est ensuite traitée sous pression après avoir été lavée à l'eau. Le traitement sous pression est réalisé avantageusement par moulage à une température comprise entre 100 et 160°C, par exemple 150°C, sous une pression d'environ 0,4 à 0,8 kg/cm^2, de préférence 0,5 kg/cm^2.

Selon une variante de mise en oeuvre du procédé, on réalise ce traitement sous pression en extrudant la fraction protéique sous pression à une température d'environ 130 à 250°C, par exemple de 150°C.

Le produit obtenu après ce traitement sous pression peut être appelé "fromage de protéines".

Selon le procédé de l'invention, on congèle ensuite le fromage de protéines à une température au plus égale à -5°C. La température de congélation peut varier entre environ -5°C et -20°C; le temps de congélation est fonction de la température de congélation; à titre d'exemple on indiquera que le temps de congélation est d'environ 24 heures à la température de -10°C.

Le fromage de protéines congelé est ensuite soumis à une décongélation à une température au moins égale à la température ambiante, de préférence à la vapeur chaude.

Le produit obtenu après décongélation constitue un aliment protéique qui possède des caractéristiques organoleptiques proches de celles de la viande.

L'aliment protéique obtenu selon l'invention peut éventuellement être ensuite aromatisé et emballé par exemple sous forme en portions alimentaires.

De plus, il faut noter que le produit selon l'invention est stable au chauffage.

Les produits obtenus selon le procédé de l'invention contiennent plus de fraction 11 S que ceux précipités en milieu acide et contiennent donc une proportion supérieure d'acides aminés soufrés compte-tenu de la richesse relative de la fraction 11 S en ces composés par rapport à la fraction 7 S (voir à cet effet SAIO K. et al. Food use of soybean 7 S and 11 S proteins - Extraction and functional properties of their fractions - J. Food Sci. 38:1139).

Les produits obtenus selon le procédé de l'invention présentent des qualités nutritionnelles supérieures aux produits actuellement sur le marché; les protéines ne sont pas dénaturées, il n'y a pas dans les aliments protéiques de l'invention d'acides aminés néo-formés; de plus, les aliments protéiques de l'invention contiennent une proportion moindre de sucres fermentescibles. Ils présentent une teneur en protéines (N x 6,25) d'au moins 60%; une teneur en glucides d'environ 3 à 10%; une teneur en cendre d'environ 4 à 6%.

Les produits selon l'invention peuvent être directement emballés après l'étape de décongélation ou séchés pour la conservation; ils sont ensuite réhydratés au moment de la consommation.

On a constaté que la croissance chez le rat nourri avec l'aliment protéique selon l'invention est satisfaisante; elle est du même ordre que celle obtenue avec d'autres produits à base de soja; le coefficient d'efficacité protéique (C.E.P.) est de 2 environ.

L'invention sera illustrée plus en détail par les exemples ci-après non limitatifs.

EXEMPLE 1

Dans cet exemple on a mélangé un kg de tourteau de soja avec 9 litres d'eau en présence de 424 grammes de Na_2CO_3 (0,4 M); on a ajouté de l'hydroxyde de sodium en quantité suffisante pour ajuster le pH du mélange réactionnel à 8; on a chauffé le mélange réactionnel jusqu'à 50°C et on a maintenu cette température pendant 2 heures sous agitation. On a ensuite filtré le mélange réactionnel;

on a ajouté au filtrat 150 ppm de $NaHSO_4$; on a chauffé le mélange réactionnel à 110°C pendant 10 minutes; on a fait refroidir le mélange jusqu'à 70°C et on a ajouté une solution de $CaCl_2$ à 25% (50g de $CaCl_2$ pour 1 kg de tourteau). Le milieu réactionnel a ensuite été décanté et filtré. La fraction protéique, c'est-à-dire le caillé, ainsi obtenue, a ensuite été moulée à chaud à 150°C sous une pression de 0,5 kg/cm^2, puis congelée à -10°C pendant 24 heures. Le produit résultant a ensuite été décongelé pendant 24 heures.

La composition du produit final, c'est-à-dire de l'aliment protéique, ainsi obtenu est donnée dans le tableau I ci-après.

EXEMPLE 2

On a traité 1 kg de graines de soja selon le procédé de l'invention.

On a fait tremper pendant 8 heures 1 kg de graines de soja dans de l'eau à 15°C en présence de 424 grammes de Na_2CO_3 0, 4 M. On a ensuite éliminé l'eau; puis on a rincé le mélange réactionnel à l'eau et on a broyé les graines gonflées (1 volume de graines/4 volumes d'eau) en présence de 150 ppm de Na HSO_4. On a ensuite chauffé le mélange réactionnel à 110°C pendant 10 minutes et on l'a filtré.

On a ajouté au filtrat, à 70°C, 50g de $CaCl_2$ sous forme d'une solution de $CaCl_2$ à 25%; on a décanté et filtré. La solubilité et la viscosité d'une telle fraction protéique ne varient pratiquement pas en fonction de la durée du chauffage à 100°C. De même, la viscosité et la solubilité de cette fraction en fonction du pH sont très faibles au niveau de pH 5.

La fraction protéique ainsi obtenue a été moulée à 150°C sous une pression de 0,5 kg/cm^2 ; elle a ensuite été congelée à - 10°C pendant 24 heures, puis décongelée à la vapeur.

La composition du produit final ainsi obtenu est donnée dans le tableau I ci-après.

La figure 1 représente la variation de la solubilité à 20°C de la fraction protéique (solution titrant 20% en protéines) en fonction du pH; en ordonnées on a porté le pourcentage en poids du sédiment soluble et en abscisses le pH.

La figure 2 représente la variation de la viscosité à 20°C de la fraction protéique (solution titrant 20% en protéines)

en fonction du pH dont les valeurs sont portées n abscisses, la viscosité en centipoises étant portée en ordonnées.

La figure 3 représente la variation de la viscosité de la fraction protéique (solution titrant 20% en protéines) en fonction du temps de chauffage à 100°C, le pH de la solution étant de 6,5; la viscosité en centipoises est indiquée en ordonnées et en abscisses on a porté le temps de chauffage en minutes.

La figure 4 représente la variation de la solubilité de la fraction protéique (solution titrant 20% en protéines) en fonction du temps de chauffage à 100°C, le pH de la solution étant de 6,5; le pourcentage de sédiment soluble est porté en ordonnées et le temps de chauffage en minutes est indiqué en abscisses.

TABLEAU I

Composition des produits protéiques obtenus selon l'invention

(Pourcentage par rapport à l'extrait sec)

	Protéine (N x 6,25)	Glucides	Lipides	Cendres
Produit final obtenu à partir du tourteau (exemple 1)	≃ 75	≃ 10	≃ 0	≃ 5
Produit final à partir de la graine entière (exemple 2)	70	≃ 10	≃ 8-10	≃ 5-6

REVENDICATIONS

1. Procédé pour l'obtention d'aliments protéiques à partir de tourteaux ou graines d'origine végétale, caractérisé en ce qu'il consiste :

1) à faire coaguler les protéines à partir d'un produit résultant d'un traitement préalable des graines ou des tourteaux d'origine végétale, ladite coagulation étant réalisée à l'aide d'un sel alcalino-terreux;

2) à traiter sous pression la fraction protéique résultante pour obtenir un fromage de protéines;

3) à porter le fromage de protéines à une température de congélation au plus égale à -5°C; et

4) à décongeler ledit fromage de protéines à une température au moins égale à la température ambiante.

2. Procédé selon la revendication 1, caractérisé en ce que le traitement préalable des graines ou des tourteaux d'origine végétale consiste essentiellement en une étape de trempage pendant 3 à 8 heures à une température comprise entre 10 et 60°C et en une étape de chauffage à environ 100 à 120°C pendant 10 à 20 minutes environ.

3. Procédé selon l'une des revendications 1 ou 2, caractérisé en ce que l'on utilise un tourteau de graines d'origine végétale et en ce que le trempage est réalisé à environ pH 8 pendant environ 3 heures à une température comprise entre 40 et 60°C environ.

4. Procédé selon l'une quelconque des revendications 1 à 3, caractérisé en ce que l'on utilise des graines d'origine végétale et en ce que le trempage est réalisé pendant environ 8 heures à une température comprise entre environ 10 et 20°C.

5. Procédé selon l'une quelconque des revendications 1 à 4, caractérisé en ce que le trempage est réalisé en présence de Na_2CO_3.

6. Procédé selon l'une quelconque des revendications 1 à 4, caractérisé en ce que le chauffage a lieu en présence d'un agent anti-oxydant, tel que $NaHSO_4$ et $Ca(OH)_2$ à 110°C pendant environ 10 minutes.

7. Procédé pour l'obt ntion d'une fraction protéique, stable au chauffage, caractérisé en ce qu'il consiste à tremper des graines ou tourteaux d'origine végétale pendant 3 à 8 heures à une température comprise entre 10 et 60°C en présence de Na_2CO_3, à chauffer le mélange résultant à une température comprise entre 100 et 120°C pendant environ 10 à 20 minutes et à faire coaguler les protéines à l'aide d'un sel d'alcalino-terreux, ladite fraction protéique étant ensuite isolée par décantation et filtration.

8. Procédé selon l'une quelconque des revendications 1 à 6, caractérisé en ce que le traitement sous pression est réalisé par moulage sous une pression d'environ 0,4 à 0,8 kg/cm^2 à une température d'environ 100 à 160°C.

9. Procédé selon l'une quelconque des revendications 1 à 6, caractérisé en ce que le traitement sous pression est réalisé par extrusion à une température d'environ 130 à 250°C.

10. Aliment protéique obtenu par le procédé selon l'une quelconque des revendications 1 à 9.

11. Aliment protéique obtenu à partir de tourteaux ou graines d'origine végétale, caractérisé en ce qu'il présente une teneur en protéines (N x 6,25) d'au moins 60%, une teneur en glucides d'environ 3 à 10% et une teneur en cendres d'environ 4 à 6%, ledit produit protéique ayant des caractéristiques organoleptiques semblables à celles de la viande.

Pl 1/4

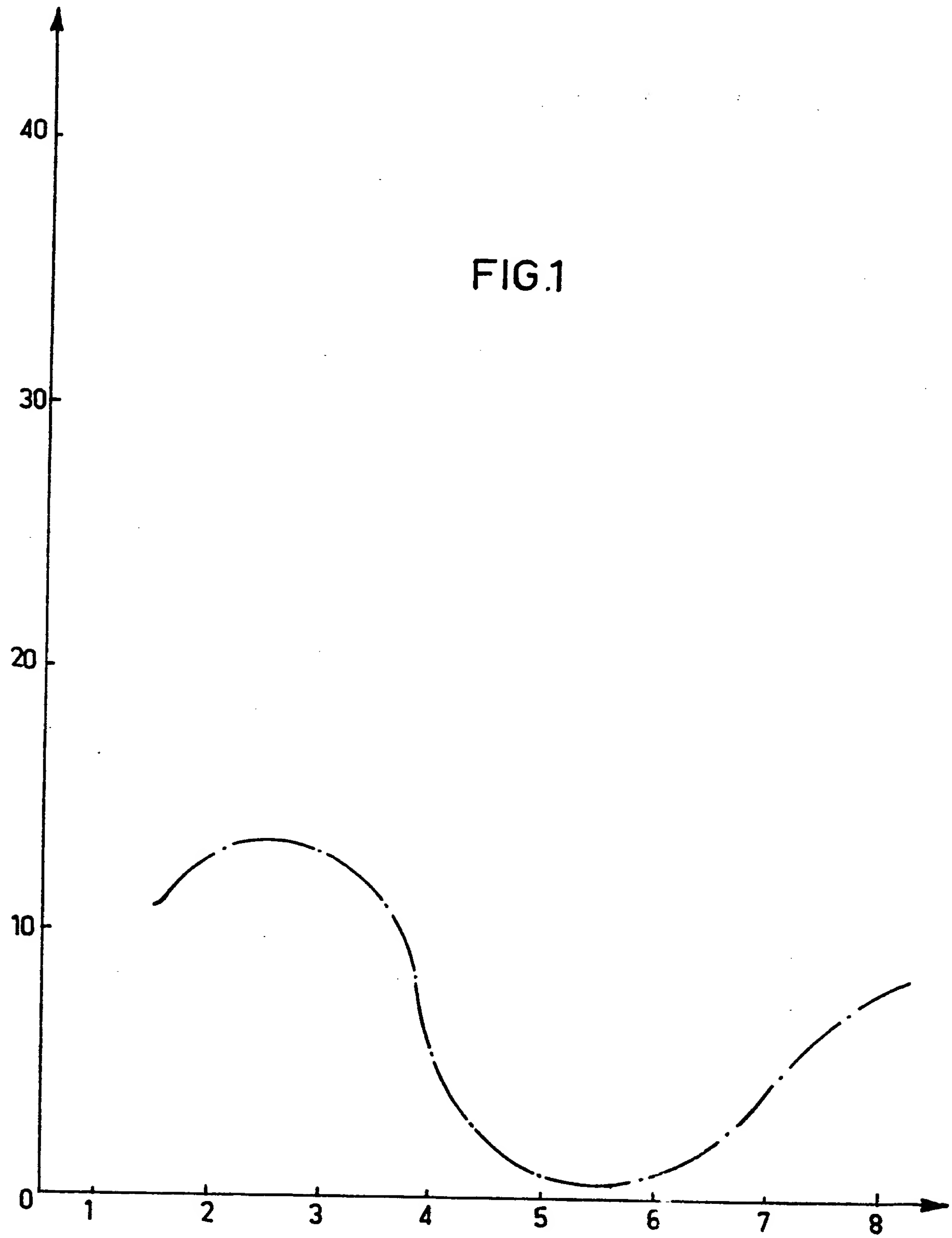
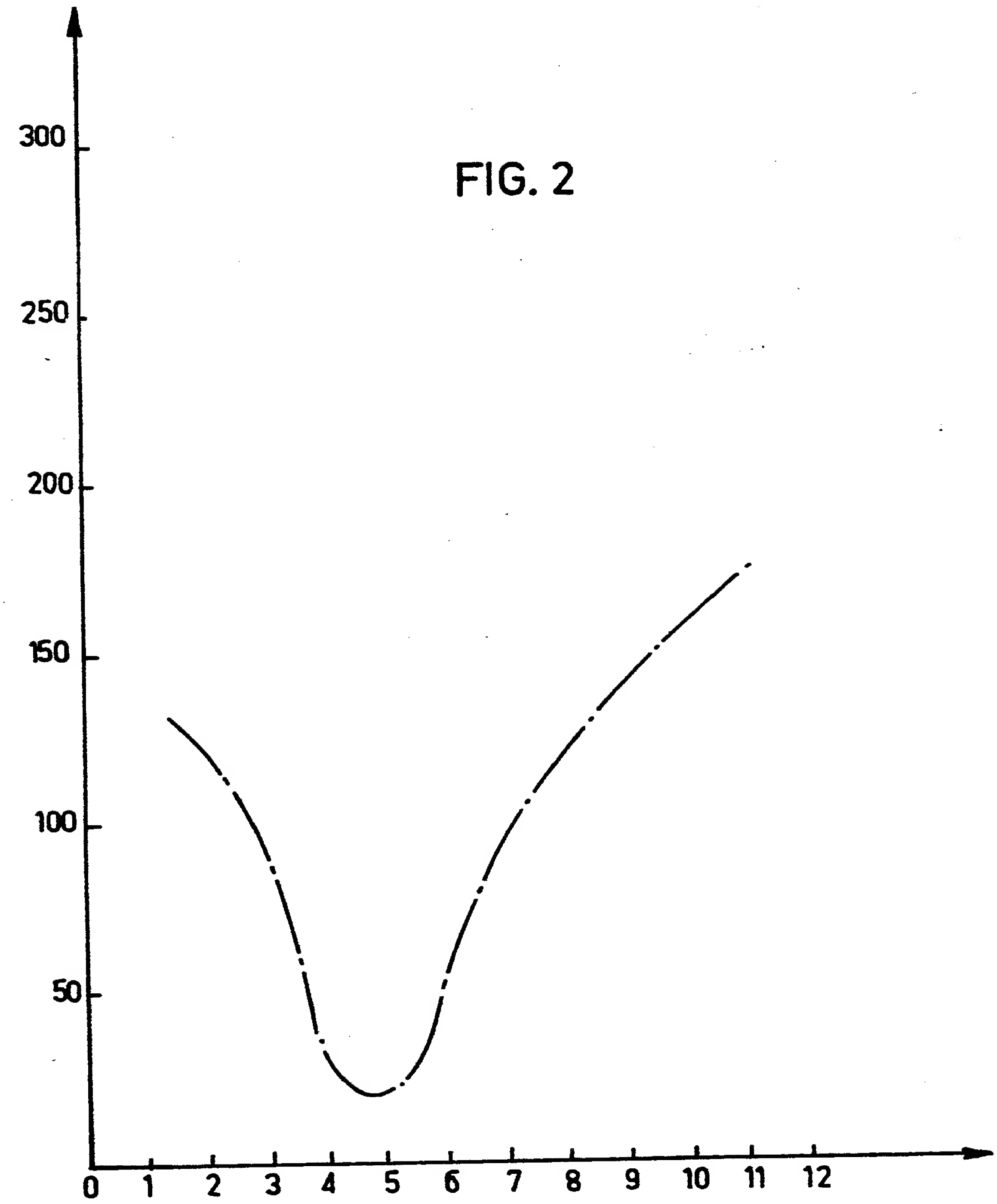


FIG.1

FIG. 2



Pl. 3/4

FIG. 3

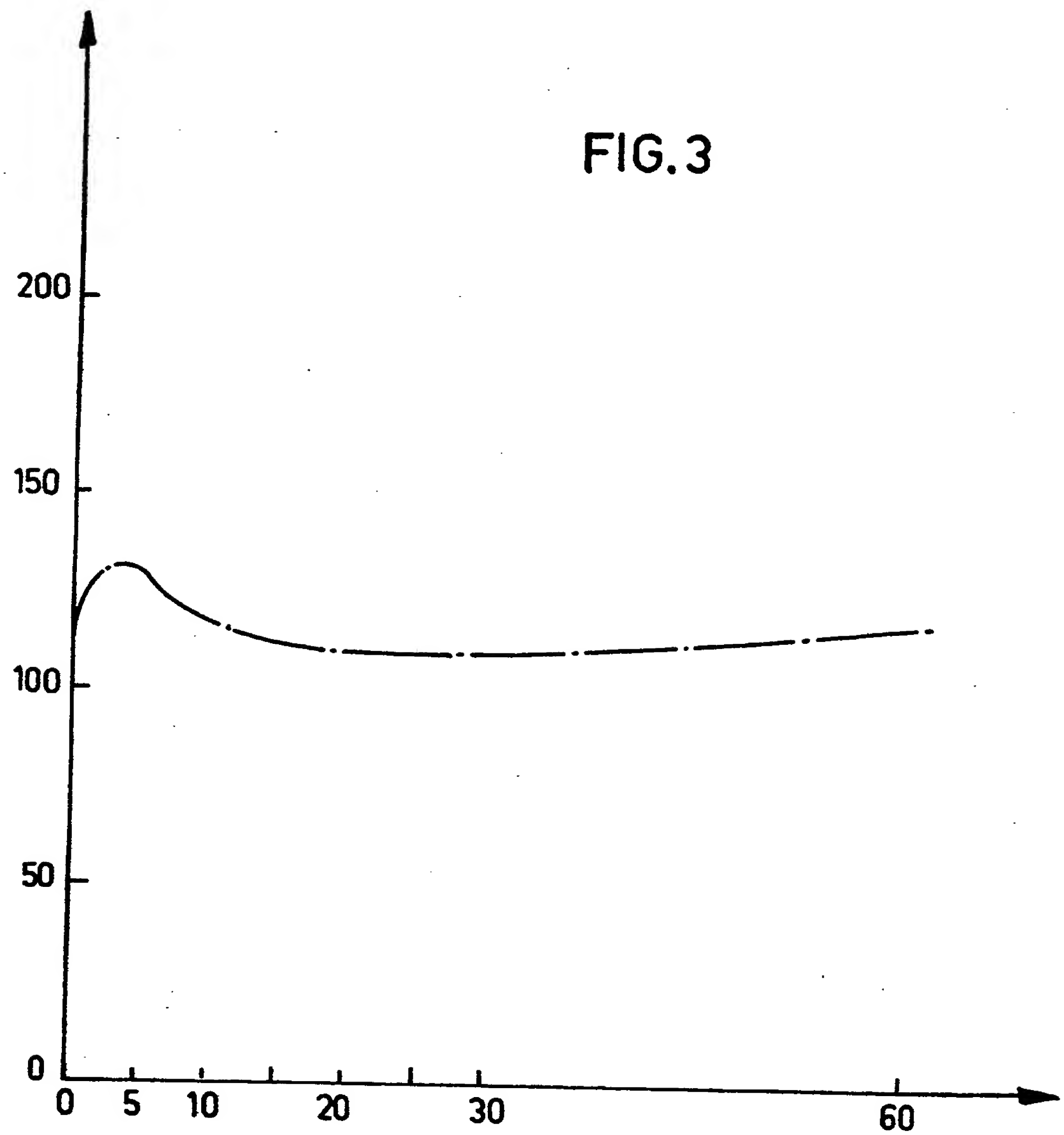
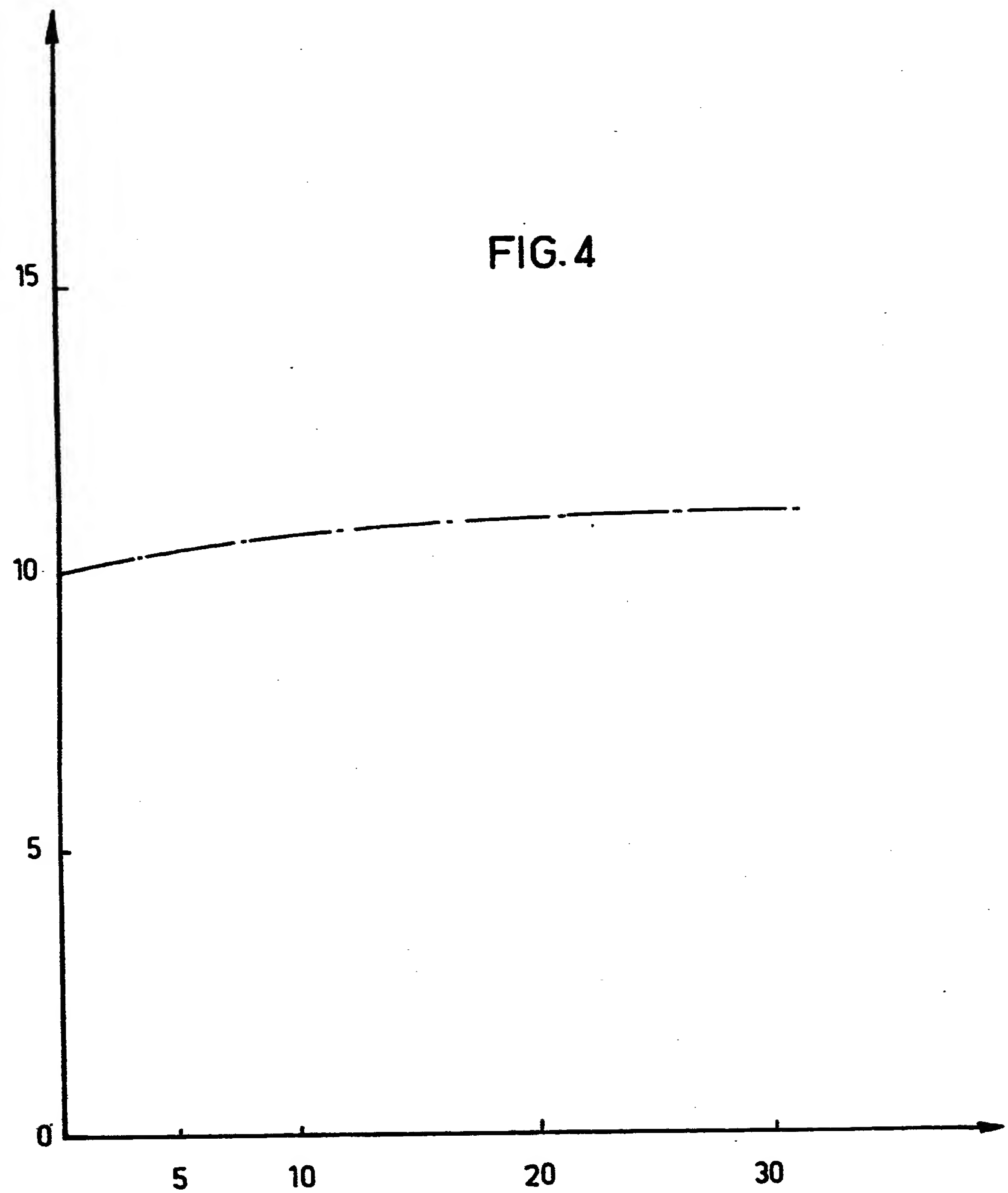


FIG. 4



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(54) Title: FORMULATED EMULSION PRODUCT AND PROCESS

(57) Abstract

A process for the production of a formulated emulsion product which has a meat-like appearance. A protein source is emulsified and then alkaline is added to the emulsion to raise the pH to above about 8. The emulsion is rapidly treated in an emulsion mill to cause the protein in the heated emulsion to at least partially coagulate. The heated emulsion is then allowed to coagulate and form striations and is cut into chuncks. The formulated emulsion product so produced contains about 45 % to 85 % by weight moisture and has a chewy texture and a high striated appearance.

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Formulated Emulsion Product and Process

This invention relates to a process for the production of a formulated emulsion product which has a meat-like appearance. The invention also relates to products so produced. The formulated emulsion products are particularly suitable for use as pet foods.

Coagulated meat emulsions, in the form of loaf-like products, are commonly used as pet foods because they are easily manufactured, readily digested by the animals, very palatable to the animals, and are readily formulated to contain necessary nutrients and trace elements. A disadvantage is that these coagulated emulsions do not have a striated and chunky meat-like appearance. However, for pet foods, a meat-like appearance can greatly enhance consumer acceptability.

A formulated meat emulsion which has a meat-like appearance is described in US patent 4,781,939. This formulated meat emulsion is produced by first forming a meat emulsion from a meat source. Dry ingredients such as dry proteinaceous materials (for example wheat gluten, soy flour), vitamins, minerals and the like are then mixed into the meat emulsion to provide a viscous emulsion. The viscous emulsion is then run through a high-speed emulsion mill in which the emulsion is rapidly heated to a temperature in the range of 102°C to 118°C. The emulsion leaving the emulsion mill is fed to a holding tube where the protein in the emulsion coagulates to form a solid emulsion product. This solid emulsion product is then formed into chunks. The chunks are highly striated and resemble natural meat chunks in appearance and texture.

The product produced by the process has been successfully used in pet foods for many years. However in recent times, mainly to reduce costs, there has been a move to increase the moisture content of the meat emulsions. However, if the amount of moisture becomes too high, the emulsion is not viscous enough to form acceptable chunks. Consequently proteinaceous materials must be added to the emulsion prior to forming into chunks.

A similar process is disclosed in US patent 5,132,137. However, in this process the viscous emulsion is heated to a temperature of 40 to 70°C in the emulsion mill; which is much lower than that in the process disclosed in US patent 4,781,939. The heated emulsion takes longer to coagulate and is therefore held in a holding tube for a longer time. The emulsion is then formed into strands and baked in an oven at a core temperature of 70 to 95°C. The patent is deficient

in specific teaching of the contents of the emulsion but it is very likely that proteinaceous components such as gluten would be needed and that the moisture content would need to be low. This is particularly so since only partial coagulation occurs in the emulsion mill.

Therefore there is a need for a process of producing a formulated emulsion product which has a meat-like appearance and texture and yet which may have a higher moisture content.

In one aspect, this invention provides a process for the production of a formulated emulsion product which has a meat-like appearance, the process comprising:

emulsifying a protein source for providing an emulsion;

adding alkaline to the emulsion to raise the pH to above about 8;

rapidly heating and comminuting the emulsion using mechanical energy for causing protein in the heated emulsion to at least partially coagulate;

allowing the heated emulsion to coagulate and form striations; and

forming the coagulated emulsion into chunks for providing a formulated emulsion product.

Surprisingly, it is found that the addition of the alkaline greatly increases the viscosity of the emulsion and permits the production of a formulated emulsion product of higher moisture content. Also, if desired, components such as gluten may be reduced in amount or omitted entirely. Further, the emulsion need not be produced from a traditional meat source; instead an animal protein source such as plasma, blood proteins, whey, or casein may be used. Consequently, a formulated emulsion product with a meat-like appearance may be produced at lower cost.

Further, it is also surprisingly found that the formulated emulsion product has much thinner and more pronounced layers giving it a highly striated and more meat-like appearance. Also the product has an improved, chewy texture.

The protein source may be of animal or vegetable origin, or a mixture of both. Conveniently the protein source may comprise at least about 80% by weight of animal protein; the remaining protein being of vegetable origin. For example, the protein in the emulsion may comprise about 95% to 100% by weight of protein of animal origin. Alternatively, the protein in the emulsion may comprise about 95% to 100% by weight of protein of vegetable origin.

The alkaline which is added to the emulsion may be any suitable alkaline which is acceptable in food. For example, the alkaline may be NaOH, KOH or

$Ca(OH)_2$, or mixtures thereof. Preferably, however, the alkaline is NaOH. Preferably sufficient alkaline is added to raise the pH of the emulsion to a range of about 9 to about 12; for example about 9 to about 11. This amount preferably results in the formulated emulsion product containing less than about 5% by weight alkaline; more preferably less than about 2% by weight.

The formulated emulsion product preferably has a moisture content of about 45% to about 85% by weight; more preferably about 55 to about 65% by weight. If necessary water may be added up to an amount of about 60% by weight to obtain this level of moisture. However, if the protein source contains sufficient moisture, the addition of further moisture is not necessary. Additional ingredients such as fats, sugars, salts, spices, seasonings, flavorants, minerals, and the like may also be added to the emulsion. The amount of additional ingredients used is preferably such that they make up about 1% to about 35% by weight of the formulated emulsion product.

The protein source may include whey. This embodiment has the advantage that sugars in the whey may react with amino acids in the emulsion. The Maillard reaction products which result provide the chunks with "meaty" flavor notes which may increase palatability.

The alkaline may be added to the emulsion during emulsification of the protein source, or may be added to the emulsion immediately prior to the heating and comminuting of the emulsion, or may be added to the emulsion during the heating and comminuting of the emulsion. Preferably, the emulsion is deaerated after emulsification.

Preferably the emulsion is rapidly heated and comminuted by forcing the emulsion between a pair of spaced-apart plates; at least one of which is rotating at high speed. If necessary, the emulsion may be further heated by injecting superheated steam into it. Preferably the emulsion is heated to a temperature above about 100°C, for example within the range of about 102°C to about 120°C. Alternatively, the emulsion may be rapidly heated to a lower temperature providing that the temperature is sufficient to cause at least partial coagulation of the protein; for example at least to about 45°C.

The heated emulsion is preferably allowed to coagulate and form striations for a time in the range of about 30 seconds to about 10 minutes; more preferably about 1 minute to about 6 minutes.

The invention also provides a formulated emulsion product produced by the process defined above.

In another aspect, this invention provides a formulated, striated emulsion product having a meat-like appearance, the product comprising protein, fats and about 45% to 85% by weight moisture and having a pH of above about 8.

Preferably the formulated emulsion product comprises about 55% to about 65% moisture. Also, the formulated emulsion product preferably has a pH in the range of about 9 to about 12.

The protein content of the formulated emulsion product is preferably about 10 to about 25 % by weight; for example about 12 to about 22 % by weight. The protein may include whey.

The formulated emulsion product preferably comprises about 5% to about 25% by weight of fat.

Preferably at least about 50% of the striations of the formulated emulsion product have a thickness less than about 50μm. Further, the formulated emulsion product preferably has an elasticity recovery of greater than about 40%; more preferably greater than about 50%. For example, the formulated emulsion product may have an elasticity recovery of about 55% to about 65%. This provides the product with an excellent chewy texture.

The invention also provides a pet food comprising chunks of a formulated emulsion product as defined above, in combination with an acidic carrier to neutralize the basicity of the chunks. The carrier may be an acidic sauce or gravy, or may be a coagulated meat emulsion.

Embodiments of the invention are now described, by way of example only, with reference to the drawing which illustrates schematically the process of producing the formulated emulsion product.

Referring to the drawing, a protein source 2, water 4 and an additive mixture 6 are mixed together in a mixer 8 to provide a primary emulsion 10. The protein source 2 which is fed to the mixer 8 may be any suitable meat or vegetable protein source, or both.

A suitable meat protein source is a meat emulsion obtained by grinding and then emulsifying blocks of a meat material, such as meat or meat by products. The meat material may be any suitable source of animal protein; for example the muscular or skeletal meat of mammals, poultry, and fish or meat by-products such as hearts, liver, kidneys, tongue and the like. The exact composition may be selected according to cost and the desired flavor. The meat material conveniently may be in frozen form prior to grinding. Suitable procedures for processing frozen blocks of meat material into a meat emulsion

mixture are described in US patent 4,781,939, the disclosure of which is incorporated by reference. Alternatively or in addition, the animal protein source may be in the form of meat meals such as poultry meal, fish meal, and red meat meal; blood proteins; egg solids; casein; plasma; whey and mixtures thereof. Again the exact composition may be selected according to cost and the desired flavor. Mixtures of any of the above may also be used.

Any suitable vegetable protein source may be used; for example grain flours and legume flours, vegetable protein isolates and concentrates, and vegetable proteins. Suitable examples are wheat flours, cottonseed meal, gluten, soy meal, soy protein concentrates and isolates, and mixtures thereof. Preferably however the vegetable protein source contains reduced amounts of starch; for example, less than about 20% by weight of starch. Cottonseed meal, gluten, soy meal, soy protein concentrates and isolates are therefore particularly preferred.

It is particularly preferred to use a protein source which is a mixture of animal and vegetable origin; for example a mixture of meat material, soy protein isolates or concentrates, and gluten. The total amount of protein in the primary emulsion must be such that the protein is able to coagulate to provide a firm emulsion product in a relatively short time; for example in less than 10 minutes. For example, the primary emulsion 10 may contain from 10 to 22 % by weight of protein.

If desired, whey may be included in the protein source 2; for example in an amount of about 1% to about 25% by weight of the primary emulsion 10. The whey is conveniently in powder form. The inclusion of the whey has the advantage of providing a source of reducing sugars which may react with amino acids to provide Maillard reaction products. These reaction products may provide flavor notes which can increase the palatability of the formulated emulsion product.

The additive mixture 6 may be a mixture of dry additives or fats; and mixtures thereof. The dry additives may be, for example, sugar, salt, spices, seasonings, vitamins, minerals, flavorants and the like. The fats may be suitable animal fats; for example tallow, or may be vegetable fats. The total amount of the additive mixture used may be selected as required but will usually be in the range of 1 to 35% by weight of the primary emulsion.

The amount of water 4 added is sufficient such that the water comprises from about 45% to 85% by weight in the formulated emulsion product. If sufficient moisture is present in the protein source 2 or the additive mixture 6, the

water 4 may be omitted. Usually an amount of water to make up to about 60% of the primary emulsion 10 is added.

In the processes described in US patents 4,781,939 and 5,132,137, the amount of fat in the primary emulsion must be controlled to facilitate processing and to obtain an acceptable product. However the amount of fat is not an important parameter in this process and the amount can be selected as desired. Consequently no fat need be added; particularly if the animal protein source 2 contains fats. Conveniently, the amount of fat in the primary emulsion 10 is in the range of 5% to 25% by weight. Reducing the importance of fat as a parameter in the process is a major advantage of the process.

The protein source 2, the water 4 and the additive mixture 6 are mixed in any suitable mixer 8. For example, a twin screw mixer, a twin ribbon blender, or an overlapping paddle mixer may be used. The mixing should be sufficient to ensure that any dry ingredients are dissolved.

Once mixed, the primary emulsion 10 is preferably fed through a vacuum stuffer (not shown), or similar deaeration apparatus, to deaerate the primary emulsion 10. This removes air which may otherwise cause disruption of the formulated emulsion product and reduce its meat-like appearance. However, it is not essential to deaerate the primary emulsion 10 because, in general, an acceptable product may still be obtained.

Alkaline 12 is injected into the primary emulsion 10 prior to the primary emulsion 10 being fed into an emulsion mill 14. Sufficient alkaline 12 is injected so as to raise the pH of the primary emulsion 10 to a range of about 8 to about 12. The alkaline may be any alkaline suitable for use in a food product. Conveniently, NaOH may be used; preferably in solution. The concentration of the NaOH in solution is conveniently about 25% to 50% by weight. As an alternative to injecting the alkaline into the primary emulsion 10, the alkaline may be added into the mixer 8 along with the water 4 and additive mixture 6. As a further alternative, the alkaline may be injected into the primary emulsion 10 as it enters the emulsion mill 14.

The emulsion mill 14 is a high speed mill which subjects the emulsion to rapid mechanical heating and shearing. Any suitable emulsion mill 14 may be used, for example the emulsion mill disclosed in US patent 5,132,137, the disclosure of which is incorporated by reference. Other suitable emulsion mills 14 are commercially available under the tradename of Trigonal and may be obtained from Siefer Machinenfabrik GmbH & Co KG, Bahnhofstrasse 114,

Postfach 101008, Velbert 1, Germany. These emulsion mills usually comprise a pair of plates which are closely spaced apart. One of the plates rotates at high speeds. The emulsion is fed between the plates and the mechanical energy imparted to the emulsion rapidly raises its temperature. The heated emulsion 16 leaves the emulsion mill in a thin stream which packs onto earlier streams to form layers or striations

The temperature of the primary emulsion 10 is raised to the desired coagulation temperature in the emulsion mill 14 in a few seconds. For example, the temperature may be raised to from about 100°C to about 120°C. Alternatively, the temperature may be raised to in the range of about 45°C to about 75°C as described in US patent 5,132,137. Usually the mechanical energy generated in the emulsion mill 14 will be sufficient to heat the emulsion 10 but this may be supplemented by the injection of superheated steam.

The heated emulsion 16 leaving the emulsion mill 14 is then transferred to a holding tube 18. Holding tubes 18 such as described in US patents 4,781,939 and 5,132,137 may be used. In the holding tube 18, the heated emulsion 16 coagulates while moving slowly along the holding tube 18. The residence time of the heated emulsion 16 in the holding tube 18 is sufficient for the emulsion to have coagulated into a firm emulsion product upon reaching the exit of the holding tube 18. Residence times of about 30 seconds to about 10 minutes are sufficient; the actual time depending upon the temperature to which the primary emulsion 10 was heated. Longer or shorter residence times may also be used but disadvantages arise. If desired, the holding tube may be equipped with a suitable valve at its end to maintain a desired pressure in it.

The firm emulsion product 20 leaving the holding tube 18 is then transferred to a cutter 22 where it is cut into chunks 24 of size suitable for use in a pet food. Alternatively, if it is desired to produce chunks of a more natural appearance, steam may be intermittently injected into the holding tube 18 to cause disruption of the firm emulsion product 20 into chunks as it leaves the holding tube 18. This procedure is described in US patent 4,781,939.

If desired, the chunks 24 may be baked by passing them through an oven as described in US patent 5,132,137. However, if the meat emulsion 10 was heated to above 100°C in the emulsion mill 14, this is unnecessary.

The chunks 24 are then allowed to cool and are collected. If desired, the collected chunks are stored for later use or are simply transferred to a canning operation. Prior to canning, the chunks 24 may be flaked if desired. In the

canning operation, the chunks are filled into cans along with a gravy or sauce. The gravy or sauce may contain an acid, such as citric acid or phosphoric acid, which is acceptable in foods to neutralize the alkaline in the chunks. The cans are then sealed and sterilized. In this case, the product produced is a chunk-in-gravy type product. Alternatively the chunks may be combined with a meat emulsion which forms a loaf-type product. In this case, the meat emulsion may contain a similar acid to neutralize the chunks. The cans are then sealed and sterilized. In this case, the product produced is a chunk-in-loaf type product.

The chunks 24 have a highly striated appearance which provides a very good simulation of meat. This greatly increases the consumer acceptability of the product. Also, the chunks 24 have a chewy texture which requires the animals to chew them; much as animals would chew fresh meat. This offers the advantage of being beneficial to the animals' teeth.

Although the invention has been described with reference to pet foods, it will be appreciated that the process may also be used to produce foods intended for human consumption.

Example 1

An emulsion is formed by mixing 20% plasma, 20% whey powder, 6% tallow, and 52% water (the percentages being based on the total weight of the chunk ingredients). The emulsion is transferred to a batch blender and 2% of a solution containing 50% by weight of NaOH is added. The pH of the emulsion increases to about 11.

The emulsion is run through an emulsion mill (a Trigonal Mill obtained from Siefer Machinenfabrik GmbH & Co KG). The emulsion leaving the emulsion mill is at a temperature of 103 to 105°C and is discharged into a holding tube. The residence time in the holding tube is less than 6 minutes. The coagulated emulsion leaving the holding tube is cut into chunks of about 10 mm size. The chunks have a highly striated, meat-like appearance. Some meat-like browning, due to Maillard reactions between amino acids in the plasma and sugars in the whey, is noticeable.

A loaf product mix is prepared from about 53% by weight of meat and meat by-products, about 44.5% water and about 2.5% flavoring agents. The mixture is cooked in a batch mixer/cooker and filled, with the chunks, into cans.

The mass ratio of loaf ingredients to chunks is about 78:22. The cans are sealed and sterilized.

A can is opened, a representative sample of the product is taken and the pH is determined to be about 6.5 to 7.0. Further product is fed to dogs and is freely eaten.

Example 2

Chunks are produced as described in example 1. A gravy is then prepared by mixing together 5.5% starch, 1% phosphoric acid, less than 0.5% condiments, and water making up the remainder (all percentages being by weight). The chunks and gravy are combined in a mass ratio of about 45:55 and filled into cans. The cans are sealed and sterilized.

A can is opened, a representative sample of the product is taken and the pH is determined to be about 6.3 to 6.6. Further product is fed to dogs and is freely eaten.

Example 3

Frozen meat and meat by-products obtained from a local source are ground and then emulsified. An emulsion mixture is then formed by mixing 2% plasma, 2% whey powder, 1% condiments and 93% emulsified meat and meat by-products (the percentages being based on the total weight of the chunk ingredients). The emulsion mixture is transferred to a batch blender and 2% of a solution containing 50% by weight of NaOH is added. The pH of the emulsion mixture increases to about 11.

The emulsion mixture is run through an emulsion mill (a Trigonal Mill obtained from Siefer Machinenfabrik GmbH & Co KG). The emulsion leaving the emulsion mill is at a temperature of 103 to 105°C and is discharged into a holding tube. The residence time in the holding tube is less than 6 minutes. The coagulated emulsion leaving the holding tube is cut into chunks of about 10 mm size. The chunks have a highly striated, meat-like appearance. Some meat-like browning, due to Maillard reactions between amino acids in the plasma and sugars in the whey, is noticeable.

A gravy is then prepared by mixing together 5.5% starch, 1% phosphoric acid, less than 0.5% condiments, and water; the water making up the remainder

(all percentages being by weight). The chunks and gravy are combined in a mass ratio of about 45:55 and filled into cans. The cans are sealed and sterilized.

A can is opened, a representative sample of the product is taken and the pH is determined to be about 6.3 to 6.6. Further product is fed to cats and dogs and is freely eaten.

Example 4

Frozen meat and meat by-products obtained from a local source are ground and then emulsified. The emulsion is placed in a blender and 10% by weight wheat gluten, 4% by weight soy protein concentrate, 3% condiments and 18 % by weight water are added to provide an emulsion mixture (the percentages being based on the total weight of all chunk ingredients). Two % by weight of a solution containing 50% by weight of NaOH is added then added and the emulsion mixture blended until uniform. The pH of the emulsion mixture increases to about 12.

The emulsion mixture is run through a Trigonal emulsion mill and discharged into a holding tube at a temperature of about 110°C to about 115°C. The holding tube is equipped with a pinch valve to maintain back pressure. The coagulated emulsion leaving the holding tube is cut into chunks of about 10 mm size. The chunks have a shredded, stringy; meat-like appearance.

A gravy is then prepared by mixing together 2.5% starch, 0.5% condiments, 0.3% hydrocolloid, and water; the water making up the remainder (all percentages being by weight on the basis of gravy ingredients). The chunks and gravy are combined in a mass ratio of about 60:40 and then about 1.75% by weight of phosphoric acid is added and blended in. The mixture is then filled into cans. The cans are sealed and sterilized.

Example 5

An emulsion is prepared by blending 8% by weight wheat gluten, 22% by weight soy protein concentrate, 0.3% condiments, 5% fat and 62.7 % by weight water (the percentages being based on the total weight of all chunk ingredients). Two % by weight of a solution containing 50% by weight of NaOH is added then added and the emulsion mixture blended until uniform. The pH of the emulsion mixture increases to about 11.

The emulsion is then formulated into chunks and added to a gravy as described in example 4.

Example 6

A chunk produced according to the process described in US patent 4,781,939 (Sample A) and a chunk produced according to example 4 (Sample 1) are frozen at -80°C. The samples are freeze fractured into sections of 10 to 15 μm and stained with Sudan III and Harris' Hematoxylin (*J. Dairy Science*; 1922, 5). The samples are then subjected to light microscopy in the frozen state.

Sample 1 is formed of thin layers or striations of protein and fat generally of thickness less than about 50 μm. Sample A is formed of less well defined layers or striations of protein and fat of thickness substantially generally greater than 50 μm. The results support the visual observation that the chunks of Sample 1 have an appearance more highly striated then the conventional chunks of Sample A.

Example 7

A dough which contains about 62.5% by weight of a mixture of turkey meat and fish is prepared according to example 4 (without the formation of chunks). The dough are labeled Sample 2. A similar dough is produced according to the process described in US patent 4,781,9394 (again without the formation of chunks). The dough is labelled Sample B. The doughs are then cut into disks of about 50mm diameter and 10 mm thickness. Twenty disks of sample 2 are prepared in this way and 20 disks of sample B.

Each sample disk is placed in a texture analyzer (TX.XT2 by Texture Technologist Corp, Scarsdale, New York, USA) on a flat disk of 80 mm. The temperature is about 10 to 15°C. A flat bottom probe of 56 mm diameter is pressed down at a constant speed of 2 mm/s on the sample disk using 10 gram force and for a distance of 6 mm. The probe is held in this position for 60 seconds. The strain at the start of the pressing (initial strain) and the strain after pressing (final strain) are determined and recorded. The percentage elasticity recovery of each disk is then determined according to the formula:

recovery % = 100 - ((final strain - initial strain)/final strain x 100)

The results are as follows:

			Sample 2				Sample B
Disk	Final strain	Initial strain	% Recovery	Disk	Final strain	Initial strain	% Recovery
1	1402	887	63.3	1	812	223	27.5
2	1475	943	64.0	2	763	208	27.2
3	838	519	62.0	3	543	136	25.0
4	443	272	61.4	4	829	233	28.1
5	308	196	63.8	5	810	213	26.3
6	610	389	63.7	6	1011	288	28.5
7	930	583	62.7	7	781	216	27.6
8	953	579	60.7	8	616	168	27.3
9	493	309	62.7	9	799	200	25.1
10	501	314	62.7	10	771	217	28.2
11	462	285	61.7	11	755	207	27.4
12	356	222	62.0	12	509	140	27.5
13	683	429	62.7	13	821	212	25.8
14	425	260	61.2	14	532	141	26.6
15	492	307	62.4	15	672	181	27.0
16	87	53	60.7	16	538	151	28.0
17	801	489	61.1	17	843	229	27.2
18	1253	771	61.5	18	695	180	25.8
19	911	567	62.2	19	682	184	27.0
20	325	194	59.6	20	524	142	27.0

The average % recovery for the disks of sample 2 is 63.3 while that for the disks of sample B is 27.0. This indicates that chunks produced from the dough of Sample 2 have a much more elastic (chewier) texture than the conventional chunks.

Claims

1. A process for the production of a formulated emulsion product which has a meat-like appearance, the process comprising:

emulsifying a protein source for providing an emulsion;

adding alkaline to the emulsion to raise the pH to above about 8;

rapidly heating and comminuting the emulsion using mechanical energy for causing protein in the heated emulsion to at least partially coagulate;

allowing the heated emulsion to coagulate and form striations; and

forming the coagulated emulsion into chunks for providing a formulated emulsion product.

2. A process according to claim 1 in which NaOH, KOH or $Ca(OH)_2$, or mixtures thereof, is added as the alkaline.

3. A process according to claim 2 in which sufficient alkaline is added to raise the pH of the emulsion to a range of about 9 to about 12.

4. A process according to claim 1 in which up to about 60% by weight, on the basis of the weight of the emulsion, of moisture is emulsified with the protein source for providing a formulated emulsion product having a moisture content of about 45% to 85% by weight.

5. A process according to claim 1 in which the emulsion is heated to a temperature in the range of about 102°C to about 120°C.

6. A formulated, striated emulsion product having a meat-like appearance, the product comprising protein, fats and about 45% to 85% by weight moisture and having a pH of above about 8.

7. A formulated emulsion product according to claim 6 which comprises about 55% to about 65% by weight moisture.

8. A formulated emulsion product according to claim 6 in which the protein comprises about 10% to about 25% by weight of the product.

9. A formulated emulsion product according to claim 6 in which at least about 50% of the striations have a thickness of less than about 50μm.

10. A pet food comprising chunks of a formulated emulsion product according to any of claims 6 to 9, in combination with an acidic carrier to neutralize the basicity of the chunks.

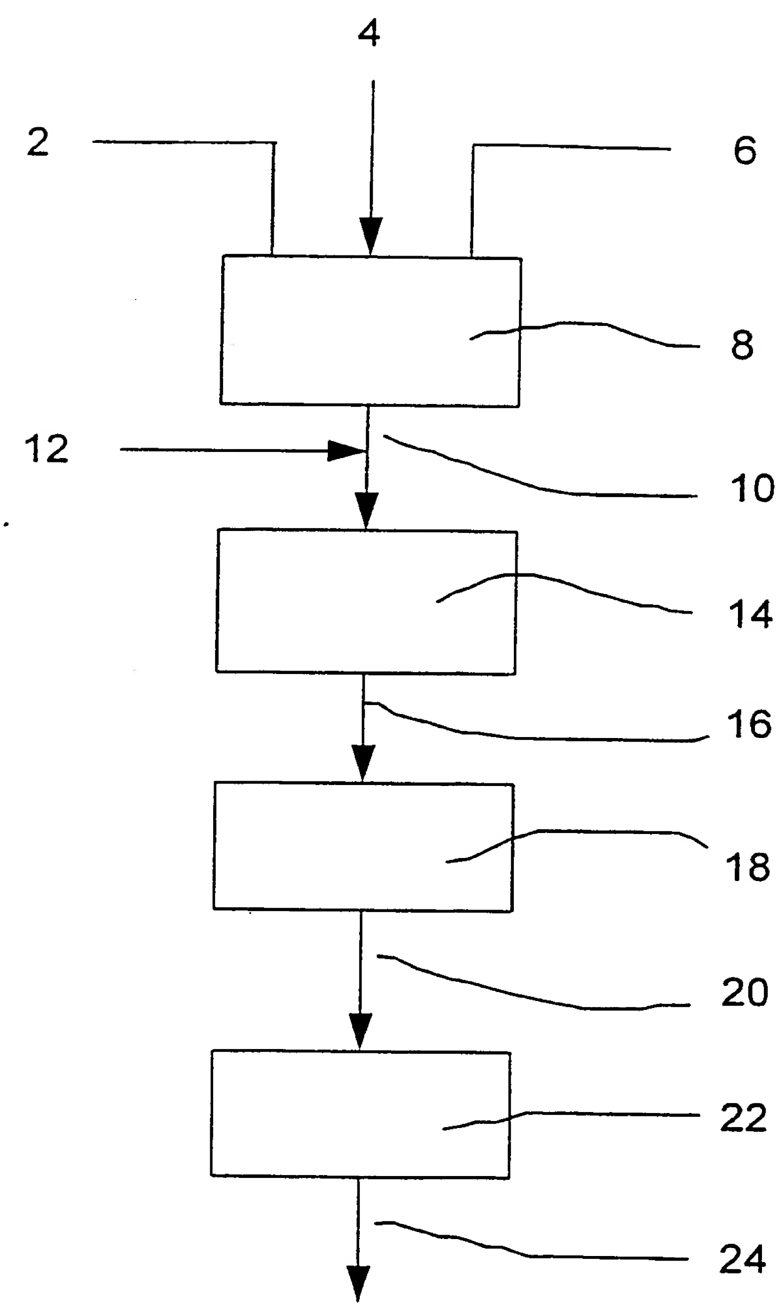

Fig. 1

INTERNATIONAL SEARCH REPORT

International Application No
PCT/EP 96/03060

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 A23L1/317 A23K1/10 A23K1/18 A23J3/22

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 A23L A23K A23J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US,A,4 781 939 (R. CRAIG MARTIN ET AL.) 1 November 1988 cited in the application see the whole document ---	1,4-8
Y	WO,A,92 18018 (NADREPH LIMITED) 29 October 1992 see page 2, paragraph 2 - paragraph 4 see page 3, paragraph 2 - paragraph 3 see page 5, paragraph 2 - paragraph 3 see example 7 see claims 1,10,14,17-20	1,4-8
A	 ---	2-4
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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

1

Date of the actual completion of the international search	Date of mailing of the international search report
7 November 1996	20. 11. 96

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax (+31-70) 340-3016	Dekeirel, M

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International Application No
PCT/EP 96/03060

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DATABASE WPI Week 7804 Derwent Publications Ltd., London, GB; AN 78-07553A XP002017903 & JP,A,52 148 652 (IKAWA M) , 10 December 1977 see abstract ---	1
A	US,A,3 843 815 (STANLEY H. REESMAN) 22 October 1974 see claim 1 ---	10
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1

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/EP 96/03060

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PATENT COOPERATION TRE[ATY]

PCT

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INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference	**FOR FURTHER ACTION** see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
39887/NSM		
International application No. PCT/GB 99/ 02111	International filing date *(day/month/year)* 02/07/1999	(Earliest) Priority Date *(day/month/year)* 02/07/1998
Applicant MARS UK LIMITED et al.		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.

[X] It is also accompanied by a copy of each prior art document cited in this report.

1. **Basis of the report**

   a. With regard to the **language,** the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

      [ ] the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

   b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :

      [ ] contained in the international application in written form.
      [ ] filed together with the international application in computer readable form.
      [ ] furnished subsequently to this Authority in written form.
      [ ] furnished subsequently to this Authority in computer readble form.
      [ ] the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
      [ ] the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. [ ] **Certain claims were found unsearchable** (See Box I).

3. [ ] **Unity of invention is lacking** (see Box II).

4. With regard to the **title,**

   [X] the text is approved as submitted by the applicant.
   [ ] the text has been established by this Authority to read as follows:

5. With regard to the **abstract,**

   [X] the text is approved as submitted by the applicant.
   [ ] the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No. ---

   [ ] as suggested by the applicant.
   [ ] because the applicant failed to suggest a figure.
   [ ] because this figure better characterizes the invention.

   [ ] None of the figures.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 99/02111

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 A23J3/12 A23K1/04 A23J3/22 A23J3/28

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 A23J A23K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier document but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
25 October 1999	26/11/1999

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31–70) 340–2040, Tx. 31 651 epo nl, Fax: (+31–70) 340–3016	De Jong, E

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Form PCT/ISA/210 (second sheet) (July 1992)

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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INTERNATIONAL SEARCH REPORT

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International Application No

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10.7

Decolouration of slaughterhouse blood by treatment with hydrogen peroxide

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Introduction

The erythrocyte fraction of animal blood, obtained after isolation/preparation of blood plasma by centrifugation, is a potential source of valuable protein. The erythrocyte fraction, called "centriblood" or "thick blood", contains about 33% protein, mainly haemoglobin. In a large slaughterhouse, slaughtering about one million pigs a year, some 290 tonnes/year of centriblood becomes available, yielding about 96 tonnes of protein. Application of this protein is seriously hampered by its deep colour; this material could only be incorporated in meat products at very low levels.

Decolouration of the haemoglobin would yield an attractive protein while its animal origin makes incorporation in meat products easily acceptable. For the decolouration of haemoglobin basically two methods are available.

The first method, described in the literature several times, involves removal of the coloured haem group from the protein by acidified acetone (1, 2, 3). This method yields an undenatured colourless protein. However, very large amounts of acetone are required and problems with residual acetone in the protein preparation may arise.

The second method involves oxidation with hydrogen peroxide. Although this method has been described in the literature (4, 5, 6), the nature of the protein obtained is unknown and the applicability of this process for centriblood is also unknown. As the oxidation process seems to be simple and the cost of decolouration would be low, we studied the oxidative decolouration of centriblood in relation to conditions and yield, and also evaluated the nature and technological properties of the protein obtained.

Experimental

Centriblood (33% protein), obtained fresh from a slaughterhouse, was treated with 3% hydrogen peroxide. The temperature was varied between 50 and 80°C, and the pH was varied from 7.1 to 5.0. Conditions allowing scale-up to a simple process were investigated. The procedure adopted was based on an article by BRAHN from 1941 (ref. 4).

Results

Reaction conditions

Rapid and proper mixing of centriblood and hydrogen peroxide is a prerequisite. To realize this, the viscous centriblood has to be diluted with about 7 volumes of water; a 3% peroxide concentration was chosen. Addition of peroxide to diluted centriblood at temperatures below 70°C causes strong and troublesome foaming. Centriblood should first be heated to 70°C. This means denaturation and flocculation of the protein. Decolouration of denatured haemoglobin by peroxide can only be completed at temperatures above 50°C. At 50°C, the reaction takes 30-60 min; at 70°C the reaction is completed within 10 minutes. Cooling down the hot (70°C) suspension of haemoglobin to 50°C before addition of peroxide, as suggested by BRAHN, is not necessary: it neither affected colour nor yield. Complete decolouration of centriblood could be obtained using 0.8 to 1.0 ml 3% peroxide per g centriblood. The peroxide should be added in one portion to the hot haemoglobin suspension.

The effect of pH of the original hot haemoglobin suspension on colour and yield of "bleached" protein is illustrated below.

pH-value		Colour of dried bleached protein	Protein yield from 30 g centriblood (g)
Before reaction	After reaction		
7.10	6.18	yellow/cream	8.67
6.80	6.00	"	8.67
6.45	5.74	"	8.67
6.05	5.38	"	8.55
5.55	5.00	brown/yellow	7.78
5.10	4.63	brown	6.76

The reaction can therefore best be performed at the original pH (7.10) of the centriblood.

Procedure for large-scale decolouration

An amount of 500 g centriblood, diluted with 3.5 l water, was heated to 70°C with vigorous stirring. With continued stirring, 50 ml of 3% hydrogen peroxide was added in one portion. Two to three minutes after addition of the peroxide, the suspended haemoglobin had become decoloured and coagulated into small beads of about 1 to 2 mm diameter. The beads could easily be collected on a filter. About 800 g of wet yellowish beads were obtained. Freeze-drying of the beads yielded 150 g protein; so 90% of the original protein was recovered.

Scale-up of the process using the conditions described above seems feasible.

Nature of the decoloured protein
The dried decoloured 'haemoglobin' was analysed and the analytical figures were compared with dried denatured haemoglobin obtained from centriblood with the same process as described above, but without peroxide treatment.

	Bleached/oxidised "haemoglobin" (%)	Denatured haemoglobin (%)
Dry matter	100	100
Protein	99	99
Ash	0.75	1.22
Iron	0.285	0.29

It appears that the "bleached haemoglobin" still contains iron. Presumably, only the porphyrin ring is opened and transformed into a bile pigment by the oxidation process.

Technological properties of decoloured haemoglobin
The decoloured material is completely insoluble in water and has a bland taste. Bleached centriblood (as freeze-dried beads) has been incorporated in a (sterilized) type of luncheon meat and in cooked sausage (Brühwurst) up to 10% replacement of the meat protein. The bleached protein has no binding properties, as could be expected, but rather behaved as an inert ingredient. The texture of the products became softer. Incorporation of bleached (yellowish-brown) "haemoglobin" resulted in a marked shift in the colour of the meat products from pink to reddish-brown and yellowish-brown. However, the acceptability of meat products in which bleached centriblood has been incorporated up to levels of 1 to 1.5%, or 5 to 10% protein replacement, would not be seriously hampered by the negative effect on texture and colour. At these levels the flavour and taste of the products are only slightly affected adversely.

Cost-price
Dried bleached centriblood would cost about DM 1.50 per kg. As processing is, in principle very simple, costs are determined to large extent by the price of the peroxide.

Toxicological saftey
Treatment of heamoglobin with hydrogen peroxide will presumably result in the formation of the bile pigment choleglobin and, very likely, in the formation of oxidized amino acids. Like in oxidized casein, methionine sulphone could well be formed (7). This compound is a growth depressant.

In a short-term feeding test (10 days) with Sprague Dawley rats, the net protein utilisation (NPU) of decolourized thick blood has been determined and was found to be similar to that of the original thick blood (which was heated to 70°C to flocculate the protein and then dried after filtration).

However, before the decolourized centriblood can be applied in meat products or other foods, its toxicological safety should be examined thoroughly by generally accepted methods. An analysis of the amino acid pattern after oxidation could precede the toxicological tests to get quick information on the type and extent of the changes that occurred.

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Tolerance to Transient Faults in Microprogrammed Control Units

p.535-546

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Key Words **— Microprogramming, Transient fault, Signature analysis, Error recovery, VLSI.**

Reader Aids **—**
Purpose: Widen state of the art.
Special math needed for explanations: Probability.
Special math needed to use results: Reliability theory.
Results useful to: CPU designers.

Summary & Conclusions **— This paper treats the detection and recovery from transient faults that cause errors in microprogrammed control units. Error detection is based upon a particular form of signature analysis, viz, the run-time computation of a value (the signature) that depends strictly on the microprogram path (the sequence) under execution. The microprogram is statically segmented (sequences are defined in a fixed way, at design time), and an anticipated signature is associated with each sequence. At run time, at the start of each sequence the computation of the signature is restarted, whereas at the end the computed signature is compared with the anticipated signature of that sequence.**

If the check is passed, the status of the control unit is saved in suitable registers, and the previously saved status is discarded. If the check fails, the last-saved status is restored (rolled back) and microprogram execution restarts from this last-saved point. The failed microprogram sequence is thus executed again (error recovery).

This error recovery technique covers (detect and recover) transient faults that corrupt the right sequencing in conventional microprogrammed units (eg, improper jumps from one sequence to another one). This technique is evaluated by determining the relation between the obtained error coverage capability and the necessary cost increases (added circuit area). The user defines the values of the number of signature-field bits.

The method is characterized by low costs; the solution can be tailored to application specific requirements by choosing the number of signature-field bits appropriate to the foreseen fault rates, fault characterization, and failure costs. The method is particularly suited t low fault rates where the probability that all the micro-instructions of a segment are affected by a fault is very low, so that the probability that the same microprogram segment must be repeated m re than once can be neglected.

1. INTRODUCTION

Microprogrammed control units have always been important both in general purpose computer architectures and in special-purpose dedicated machines, since through the extensive use of programmable and structured logic circuits (ROMs and PLAs) they allow for design flexibility and fast prototyping, for compatibility at instruction set level among different machines, and for structure regularity and VLSI implementation.

Acronyms

CPU	central processing unit
LFSR	linear feedback shift register
PLA	programmable logic array
PSA	parallel signature analyzer
ROM	read only memory
SF	signature field
TMR	triple modular redundancy
VLSI	very large scale integration

Other, standard notation is given in ''Information for Readers & Authors'' at the rear of each issue.

We consider the introduction of fault-tolerance capacities in microprogrammed control units. This characteristic is always useful and becomes a fundamental requirement whenever microprogrammed control units are applied in critical environments (eg, embedded systems or mission-critical applications).

First, self error detection is analyzed, the aim being the detection of the widest class of errors affecting microprogrammed control units, and generated by both permanent or transient faults. Second, the possibility of recovering from errors caused by transient faults is discussed. In other words, our recovery technique is limited to the overcoming of removable errors, defined as erroneous machine states resulting from transient faults, viz, from temporary environmental conditions or temporary wrong behavior of circuits. The self-check method allows also for the identification of permanent errors, defined as erroneous machine states produced by permanent faults, viz, by irreversible causes that continue to generate these errors when processing is repeated.

Error statistics [1] emphasize that transient faults are by far more likely than permanent ones. This becomes increasingly true as VLSI technologies are pushed to higher device densities, characterized by very low power signals and then by lower immunity to noise. Moreover, mission-critical applications often relate to noisy environments [2-4]. Thus, architectures capable of identifying the presence of errors and allowing for the recovery of removable errors are attractive and ultimately lead to higher device availability.

Our error recovery technique derives from the backward error recovery software approach [5,6] usually introduced at higher levels in system architectures. A full understanding of its application at the level of microprogrammed control units

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(and the computation of its properties as well) requires an explicit reference to the architecture. For this reason, section 2 — a) discusses a reference model of architecture of microprogrammed control units, and b) presents the basic schemes for error detection and recovery.

Section 3 shows how signature analysis can be used for error detection, how error recovery can be applied to microprogram execution, and how microprograms must be modified to support this technique.

The efficiency of our method is then computed, by determining the error coverage and the safeness against transient faults as a function of circuit increase. Since added circuits also influence production yield and reliability [7, 8], these two parameters are evaluated, even if they are not directly related to transient faults.

2. TOLERANCE TO TRANSIENT FAULTS

This section treats tolerance to transient faults that affect sequencing in microprogrammed control units. We refer to the classic microprogrammed control unit initially defined by Wilkes [9] and further specified, eg, in [10]. By executing sequences of micro-instructions, this control unit fetches and executes, one after the other, machine instructions stored in a main memory. The micro-instructions are binary words containing microorder bits, viz, binary command signals activating the functional units of the processing system and managing data transfers and next address bits, that identify the next micro-instruction to be executed. Robustness against faults in the microorder bits can be achieved through coding the problem of faults affecting the micro-instructions sequencing is much more complex, and is addressed here.

The functional units operating upon data are external to the control unit. The control unit has the microprogram store (a ROM in our case) containing micro-instructions addressed through a microaddress decoder and the micro-instruction address generator (sequencer) that computes the address of the next micro-instruction, and sends it to the microaddress decoder.

The sequencer can support conditional and unconditional jumps, straight-line sequencing, jumps to micro-subroutines and related returns. Conditions for conditional jumps are provided by flag signals that are generated by the Arithmetic-Logic Unit and by other external units. The micro-instruction sequencer selects the address of the next micro-instruction by using:

- a microprogram counter, incremented during each micro-instruction execution (for straight-line sequencing)
- the next address bits of the micro-instruction currently executed (for micro-jumps and micro-subroutine calls)
- a micro-stack (for return from micro-subroutines), where the microprogram counter is saved during micro-subroutine calls
- an operation-code decoder (for the start of a new micro-instruction sequence). At the start of the execution phase of a machine instruction, this circuit receives the operation code of the fetched machine instruction and translates it into the address of the first micro-instruction of the corresponding micro-instruction sequence.

Failures in the control unit can cause errors that produce sequencing errors (erroneous selection of the next micro-instruction to be executed). Sequencing errors can be identified only by keeping track of the executed micro-instructions sequence and by comparing it with the anticipated sequence, as deduced from both microprogram structure and actual sequencing conditions. Our approach to overcome removable sequencing errors requires the system to:

1. Detect whether a sequencing error has appeared
2. Identify the most recent point (recovery point) safely passed without errors, and for which the control unit status has been saved
3. Roll back to such recovery point by restoring the corresponding status, and then resume nominal operation.

Errors are detected by adopting the parallel signature analysis technique [11-13] suitably modified to enhance error coverage as related to microprogram structure. To recover an error, we apply the backward error recovery technique at the microprogram level by suitably modifying the structure of the control unit and the execution flow of the microprogram itself. The resulting structure requires a low hardware overhead with respect to other approaches [14], though leading to an augmented error latency.

2.1 *Basic scheme for error identification*

Signature analysis is based upon polynomial division in a binary finite field, using a LFSR as a divider by a fixed divisor polynomial. Starting from an input sequence of bits (the coefficient bits of the dividend polynomial), the LFSR computes the bits that are the coefficients of the remainder polynomial. These remainder bits (given by the status of the flip-flops of the LFSR at the end of integer polynomial division) are defined as the signature of the input bit sequence (figure 1 shows an example of an LFSR).

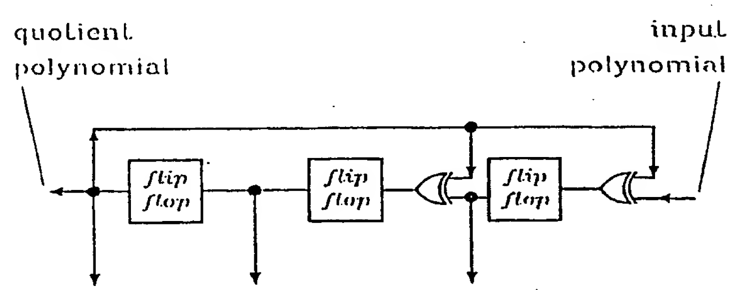

Figure 1. LFSR with Divisor Polynomial $x^3 + x + 1$

If errors are present in the input sequence, there is a possibility that the computed signature is different from the anticipated one. On the other hand, different input sequences can produce the same signature. When a particular distribution of errors transforms an input sequence into a different one characterized by the same remainder, that distribution of errors goes undetected.

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This serial technique is modified to adapt it to the architecture of the microprogrammed control unit; the first change consists in adopting a parallel signature analysis [11]. In this approach, a sequence of words of equal length is used as input, instead of a bit-serial sequence. These words are sequentially fed into a PSA — an LFSR modified to accept a whole word, in parallel, as input. Section 4.2 shows how the error detection properties of a PSA are related to those of a bit-serial LFSR characterized by the same divisor polynomial, and how the length of the signature (the degree of the divisor polynomial) influences the error-detection properties.

In order to use a PSA to keep track of a sequence of micro-instructions, a signature field is added to each micro-instruction; this signature field is fed into the PSA when the micro-instruction is executed. Consequently, the PSA changes the value of the signature accumulated into itself.

The signature computed at run-time can be compared with the anticipated one, that should be statically pre-computed and stored, at few check points, whose choice is treated in section 3.2. The anticipated signature values can be computed by a static analysis of the microprogram structure. The microprogram sequence between two consecutive check points is called microprogram segment. The micro-instruction at a check point is flagged by an added control bit which activates the check circuits.

2.2 *Basic scheme for error recovery*

If, at a check point, the anticipated and the actual signatures agree, then sequencing is assumed to be correct. Thus: all actions performed since this point are accepted, the present status of the control unit is considered correct, the PSA is reset, and nominal execution resumes with the same checking on the next microprogram segment.

If the check fails, a sequencing error has occurred. (For the moment, assume that the PSA is always fault-free; section 4.2 notes that this assumption does not limit error coverage.) A retry phase for error removal is undertaken, since, for transient faults, it is reasonable to assume that the error source disappears after a short time interval; a repetition of the last, incorrect microprogram segment would then behave correctly.

To implement this retry phase, it is necessary to restart the computation from the last known error-free status of the control unit, viz, the one assumed by the control unit corresponding to a correctly-passed check point. Obviously, to reduce error latency, the roll back phase should be minimized; for this reason, at each passed check point, the status of the control unit is saved as the recovery point for the next microprogram segment. Thus, each check point becomes a recovery point; at this check/recovery point, the following actions are performed:

a. The correct sequencing is verified by checking the PSA status after having processed the signature field of the check/recovery micro-instruction (to ensure that such check/recovery micro-instruction is the correct termination of the last microprogram segment)

b. If the check is passed, the status of the control unit must be saved before executing the check/recovery micro-instruction. Transient faults affecting execution of this micro-instruction could result in a sequencing error of the new microprogram segment being initiated, and the subsequent roll back has to re-execute this check/recovery micro-instruction to correct the error. Status saving requires that the contents of all registers of the control unit that control microprogram execution (eg, micro-address register, and micro-stack), as well as of all CPU registers meaningful for control unit status (see section 3.2), are saved to create the recovery point and, finally, the PSA is reset;

c. If the check fails, the recovery action (rolling back to the execution phase of the last correctly passed check/recovery micro-instruction) is performed.

From action b, the execution of a check/recovery micro-instruction requires two subsequent phases: 1) for signature field processing and for PSA checking, and 2) for actual execution. For all other micro-instructions, the signature field processing and the actual execution can be performed in parallel.

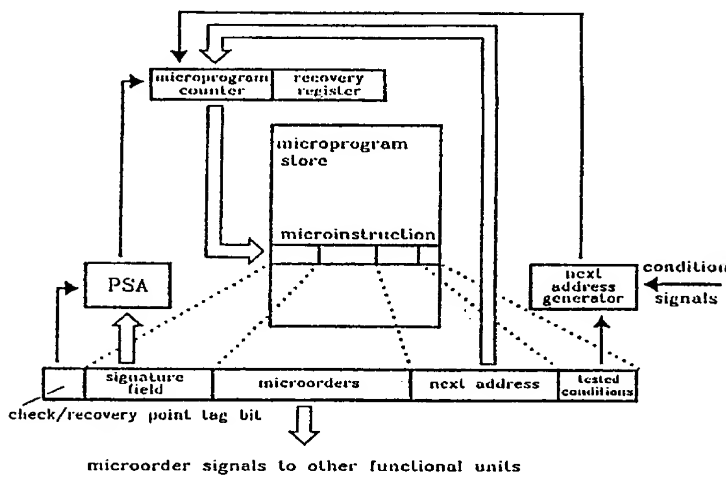

Figure 2. Augmented Architecture of the Control Unit

The above leads to definition of an augmented architecture of the control unit, as outlined in figure 2. The overall fault tolerance method thus involves (as basic component of cost) the added circuitry consisting of the PSA, of the circuits that check the signature, of the signature field added to each micro-instruction, and of the recovery registers, necessary to store the status of the control unit at recovery points. We neglect the loss of performances due to increased execution time, caused by repetition of microprogram segments in case of errors, in view of a reasonably low error rate.

3. INFLUENCE OF MICROPROGRAM STRUCTURE

This section discusses in detail the problems related to signature analysis applied to typical microprogram structures, and defines possible criteria for insertion of check and recovery points.

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3.1 *Definition of signature fields*

As outlined in section 2, checking is performed on predetermined sequences of micro-instructions (microprogram segments) bounded by recovery and check points. To allow easy checking, rather than store separately the signatures corresponding to all the check points, we prefer to define a known common signature (eg, all zeros) that the PSA should produce corresponding to all the check points.

To define signature fields for each microprogram segments, 4 cases are considered.

Case 1. The simplest case is straight-line sequencing (microprogram segment without any branch micro-instruction). In a PSA it is always possible, starting from a given remainder, to obtain any other remainder in a single step by suitably choosing the input field. Thus in a straight-line sequence of n micro-instructions, $n-1$ can have a signature field freely defined, while the last one requires a signature field that generates the common signature in the PSA.

Case 2. Wherever the microprogram segment is not constituted by a straight-line path, it becomes necessary to exploit the possibility of freely defining signature fields to differentiate as much as possible the signatures assumed inside the various microprogram paths, while ensuring that the last micro-instruction reaches the same common signature, regardless of the executed path. By applying the rules in cases 2a & 2b it is also possible to compute signature fields for microprogram loops and microsubroutines.

Case 2a. Several microprogram paths merge, as micro-instruction D in figure 3a, which is the merge node [15], viz, the common destination of the branches originating in micro-instructions A, B, C respectively.

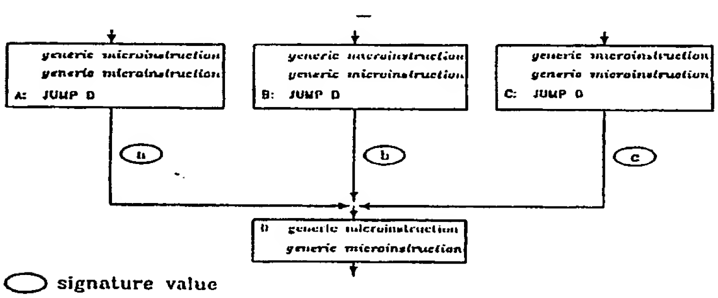

Figure 3a. Flow Diagram of a Merge Node

The three signatures a, b, c that are computed when A, B, C micro-instructions are executed are different since the different configurations derive from different paths. Yet, from all the three signatures, it is necessary to move toward a common final signature d corresponding to the final destination micro-instruction D. This implies that the signature fields of micro-instructions A, B, C have to be suitably predetermined, and cannot be arbitrary.

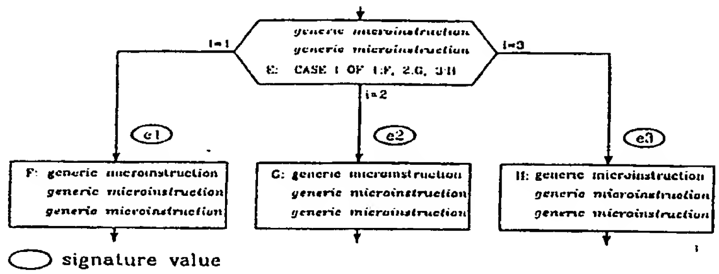

Figure 3b. Flow Diagram of a 3-Way Conditional Branch

Case 2b. A given micro-instruction E is an h-way conditional branch, after which h alternative microprogram paths can be chosen for execution, depending on external signals coming to the sequencer (figure 3b). Due to the fact that signatures depend only upon the signature fields of the executed micro-instructions, and not upon the signals that condition the jump, this signature analysis scheme is incapable of detecting sequencing errors due to faults in circuits controlled by condition signals (viz, errors leading to choice of a wrong alternative microprogram path). To overcome this difficulty the following extension of the signature analysis scheme is adopted.

The chosen signature length is increased by $\log_2(h)$ bits, being h the number of possible ways of the conditional jump. Correspondingly, also the parallel input to the PSA and the length of the signature fields are increased by $\log_2(h)$ bits. When conditional jump micro-instructions are executed, the added $\log_2(h)$ inputs of the PSA are fed with the values that the corresponding condition signals have at that execution time. Referring again to figure 3b, the signatures e1, e2 and e3 become different, allowing a check, corresponding to micro-instructions F, G, H, if the sequencing operated correctly. In all other micro-instructions, the added inputs to the PSA are the corresponding extra bits added in the signature field, whose values are freely and statically defined as usual.

Case 2c. This case arises from cases 2a & 2b for a conditional jump micro-instruction, when one of its destinations is a merge mode. As shown in case 2a, every micro-instruction preceding a merge node must be capable, by means of its signature field, to force the common signature value of the merge node. On the other hand, this cannot be possible for a conditional jump micro-instruction, because only part of its signature field can be freely defined ($\log_2(h)$ bits being the actual condition signals — see case 2b). This problem can be overcome by inserting a dummy micro-instruction in the micropogram path connecting the h-way conditional jump with the merge node. The only important part of this micro-instruction is its own signature field, statically computed to reach the predefined merge node signature (micro-order bits of the dummy micro-instruction correspond then to a no-operation).

Case 3. In microprogram loops, that have to be executed repeatedly for a variable number of times (depending on actual data values), the first micro-instruction of the loop can be considered as a merge node, requiring the definition of a signature

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common to all possible incoming paths. Since these paths correspond to the path entering the loop and to all various iterations of the loop itself, the signature fields of the loop have to be chosen so that the last micro-instruction of the loop forces the same PSA status as the path merging into the first micro-instruction, independently of the number of iterations.

Case 4. For micro-subroutines, the first micro-instruction of the micro-subroutine itself can be considered as a usual merge node, accessed by the various call points in the microprogram, while the return micro-instruction can be interpreted as a multiple-way jump without conditions. This means that, returning from a micro-subroutine, the value of the PSA status is always the same, independent of the calling point.

3.2 *Positioning of check/recovery points*

The approach in section 3.1 ensures that, at any point in a microprogram, the PSA status (signature) can be guaranteed to reach a known value, ie, positioning of check/recovery points is a-priori arbitrary, as far as checking is concerned. Positioning criteria arise from the requirements of the recovery procedure.

The information uniquely identifying the machine status — necessary for a subsequent roll back — constitutes the recovery image. Since this image must be stored in suitable recovery registers for subsequent retrieval, its extent must be restricted in order to limit the set of recovery registers. On the other hand, any such restriction leads to the presence of information that cannot be safely recovered and, therefore, leads to a decrease in the fault-tolerance capacity. For example, whenever a write operation on memory is performed, the previous contents of the affected memory location are destroyed; yet, to make the action recoverable, a full memory duplication would be required, and this would be in most cases unacceptable.

We can associate, with a given set of recovery registers, a partition of the micro-instruction set into two classes:

1. All micro-instructions acting only upon registers that are duplicated
2. All micro-instructions modifying also the contents of non-duplicated registers.

When a check point is reached, if the check is negative then the effects of the micro-instructions of class #1 can be undone simply by rolling back to the most recent recovery point; but the effects of the micro-instructions of class #2 constitute permanent (non recoverable) actions so that even a transient fault generates a permanent error. As a consequence, to protect non recoverable actions, it is necessary to associate a check/recovery point to each micro-instruction of class #2, to ensure correctness of these actions from a sequencing point of view.

It follows that check/recovery points inserted to protect the micro-instructions of class #2 are the only ones that are absolutely necessary to ensure correct sequencing of the system. Since the presence of check/recovery points leads to speed decrease (due to the 2-phase execution of check/recovery micro-instructions), it is advisable to limit the number of non recoverable actions, which implies increasing the number of resources provided with recovery registers. For this reason, we assume complete duplication of all control-unit and CPU registers.

However, restricting check/recovery points to non recoverable actions may lead to situations where error latency (time interval between appearance and detection of an error) is not acceptable, because too many micro-instructions (possibly corresponding to a few machine instructions) have to be repeated in case of error. As a consequence, excessive error latency can lead to excessive slow-down of system operation.

Error latency is not the only problem arising if too many micro-instructions are executed without any check. If the length of the input stream to a given signature analyzer increases, the probability of detecting possible errors decreases, since the number of different input streams having the same remainder obviously increases. Thus, the detection capability of this method decreases with the number of processed signature fields. For these last reasons, the insertion of additional check/recovery points is necessary.

4. ERROR COVERAGE

The probability of detecting errors by means of signature analysis depends on the characteristics of the error distribution envisioned, on the lengths of the signature field and of the microprogram segment between two subsequent check/recovery points, and on the coefficients of the divisor polynomial.

For this last item there is good and ample literature [16]. We limit ourselves to recalling that it is fairly simple to define polynomials guaranteeing detection of all single errors, all odd numbers of errors, and all double errors up to very relevant lengths of microprogram segments.

Before discussing the error distributions and quantitatively evaluating the probability of error detection, we summarize the operation performed by the PSA.

4.1 *Data compression performed by the PSA*

As shown in [11], a PSA containing s flip-flops, that processes n signature fields (corresponding to a microprogram segment of n micro-instructions) each of s bits, gives exactly the same remainder that is determined by a bit-serial LFSR (characterized by the same divisor polynomial) when this LFSR receives as input a bit stream of length $s+n-1$ obtained by computing the modulo-2 sums of all the signature fields, reciprocally shifted by one place. Figure 4 shows this SF parallelogram; every row contains a SF that is input in parallel to the PSA, and the last row shows the modulo-2 sum which is the corresponding serial input for the equivalent LFSR.

The analytic study of parallel signature properties shows that the global data compression performed by the PSA can be interpreted as equivalent to the following two subsequent steps:

1. From ns input bits (allowing 2^{ns} configurations), the SF parallelogram produces $n+s-1$ bits, thus compressing the input by a factor $2^{ns-(n+s-1)}$

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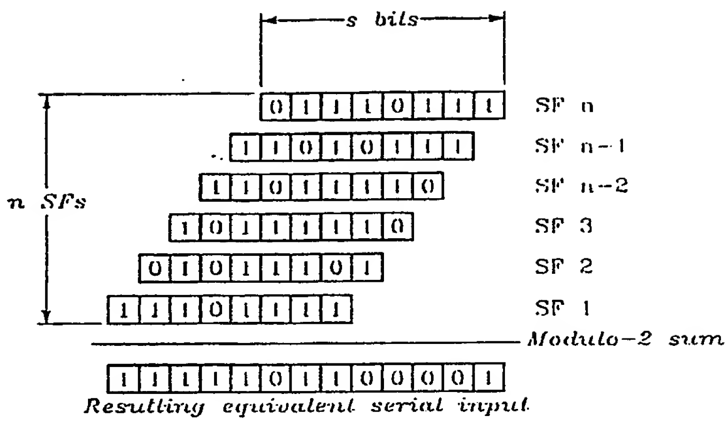


Figure 4. Example of a SF Parallelogram

2. The resulting serial equivalent input is further compressed by polynomial division, which produces a remainder of s bits, given by the final status of PSA flip-flops. This corresponds to a new compression factor 2^{n-1}.

Referring to the equivalent serial signature analysis, only 2^s remainders are possible as a response to the 2^{n+s-1} input streams (polynomials); thus several input streams can map onto one remainder. The numbers of input polynomials mapping onto the various remainders are equal; this means that onto each different remainder always map exactly —

$$2^{n+s-1}/2^s = 2^{n-1} \tag{4-1}$$

serial equivalent polynomials.

It is possible to prove the validity of this property when considering the overall sequence of signature fields processed by the PSA. The SF parallelogram in figure 4 shows that, given all possible 2^{ns} configurations, again an equal number of them ($2^{ns}/2^{n+s-1}$) maps onto each of the 2^{n+s-1} possible serial equivalent polynomials, because the modulo-2 sum of each column of the parallelogram gives either 0 or 1 for an equal number of configurations.

In conclusion, consider the 2^s input classes corresponding to the different PSA remainders, and assume that:

1. These classes are characterized by uniform density.
2. As a consequence of errors, any input sequence belonging to a given class has equal probability of being transformed into any other input sequence. □

Then, the probability that the erroneous sequence belongs to the same class of the original input sequence is given by the reciprocal of the number of input classes, 2^{-s}. Since in that case the remainder would still be the anticipated one, the probability that the signature analysis fails to detect the error is also 2^{-s}.

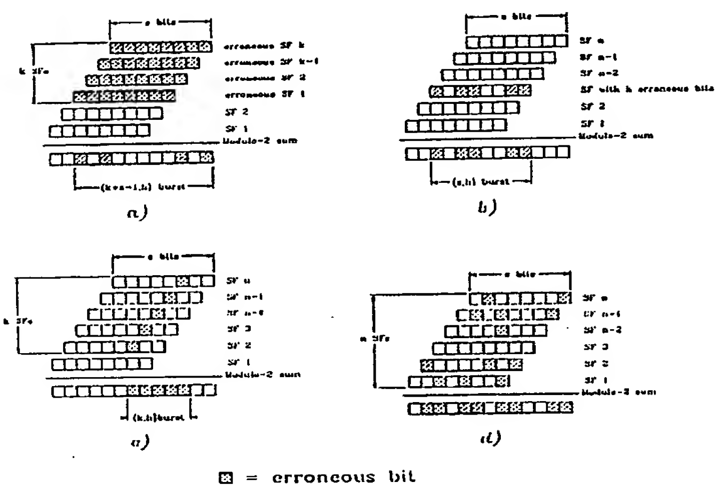
Ref [17, 18] are a detailed treatment of signature analysis coverage properties.

4.2 *Mapping of errors onto signature fields: Error coverage*

To give a detailed evaluation of the error coverage granted by our method, we need to examine how sequencing errors map in the signature fields stream that is fed into the PSA. Since the PSA is associated with an equivalent bit-serial LFSR, this section analyzes the following classes of erroneous modifications of the signature fields parallelogram.

Class 1. An unanticipated (erroneous) sequence of signature fields.

This is the case whenever failures affect the circuits that generate the next micro-instruction address (eg, next-address bits, address generator, ROM decoder). The resulting errors map into the parallelogram as shown in figure 5a.



5a. Erroneous sequencing
5b. Isolated eroneous SF
5c. Error affecting a sequence of SFs
5d. Erroneous bits randomly distributed

Figure 5. Mapping of Errors in the SF Parallelogram

Notation

k	$(0 < k \le n)$ the length of the erroneous signature fields sequence; ie, the number of unwanted micro-instructions executed before encountering a check/recovery point
n	average length of a microprogram segment
h	$\le k+s-1$ the actual number of incorrect bits; ie, the number of incorrect bits after the modulo-2 sum in the parallelogram

The corresponding error configuration in the bit-serial LFSR input is a $(k+s-1,h)$ burst, viz, a sequence of h correlated erroneous bits of maximum length $k+s-1$. The polynomial error $E(x)$ due to this type of burst can be expressed as:

$$E(x) = e(x) \tag{4-2}$$

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Notation

$e(x)$ burst error, of degree $k+s-2$.

If $k+s-2 = s-1$, (only one signature field is erroneous) the error polynomial has the same degree of the PSA remainder polynomial, that is one less than the degree of the divisor polynomial $G(x)$. This implies that $E(x)$ cannot be divided by $G(x)$ and thus it must modify the remainder. For this reason, the sequencing error is always detected.

If $k+s-2 > s-1$ then $E(x)$ passes undetected if it contains the PSA divisor polynomial $G(x)$ as a factor: the probability of this case [11] is:

$$(2^{k-1}-1)/(2^{k+s-1}-1) \tag{4-3}$$

which, when $k+s-1 \gg s$ $(k \gg 0)$, can be approximated by 2^{-s}.

Class 2. An isolated erroneous signature field.

Typical errors are the reading of a micro-instruction different from the wanted one, due to noise in the micro-address decoder during the reading phase of the microstore, or the erroneous reading of the correct micro-instruction, due to transient noise hitting the signature field of the micro-instruction itself. Errors are located randomly in single bits, or appear as burst errors whose maximum length is that of the signature fields.

Only one row of the parallelogram contains erroneous bits. As a consequence, all these bits map in corresponding erroneous bits into the equivalent serial stream, with no masking. Thus, the serial stream contains a (s,h) burst: a burst of maximum length s where h bits are incorrect. This reflects on the serial equivalent input as a polynomial error of the form:

$$E(x) = e(x) \times x^j \tag{4-4}$$

Notation

$e(x)$ burst error polynomial, of degree $s-1$
j $(0 \le j < n)$ position of the burst inside the serial equivalent input (see figure 5b).

The position of the burst polynomial error $e(x)$ does not influence error coverage [16]. This coverage depends only on $e(x)$; since $e(x)$ has the same degree $s-1$ of the PSA remainder, this class of bursts is always detected by the PSA.

Class 3. A sequence of k signature fields containing an erroneous bit in a fixed position.

Examples are those hitting a data line in the microprogram store, lasting for a time longer than that of a micro-instruction.

This class corresponds, in the parallelogram, to a column burst, where each signature field contains a single erroneous bit, and these erroneous bits are situated along a line parallel to the skewed sides of the parallelogram itself (see figure 5c).

These erroneous bits do not mask themselves, and thus cause a burst (k,h): a burst of maximum length k, $0 < k \le n$, (k is the number of affected micro-instructions in a microprogram segment) in the sequential stream. This corresponds to the error polynomial (4-4); the notation is interpreted as —

Notation

$e(x)$ burst error polynomial, of degree $k-1$
j $(0 \le j < n+s-k-1)$ position of the burst

As in class 2, the position of the burst does not influence the error coverage. This means that, if $k \le s$ then detection is ensured; else $e(x)$ can pass undetected if $e(x)$ contains $G(x)$ as a factor. This probability can be approximated by 2^{-s}.

Class 4. Erroneous bits randomly distributed in the parallelogram (figure 5d).

This is the case, eg, of repeated failures of class 2 or of failures similar to those of class 3, but hitting more than one bit in each signature field. This class of errors has been rigorously analyzed [19], with the aim of computing the exact value of signature analysis coverage of various numbers of random errors. This evaluation involves two subsequent steps.

1. Consider how random errors mask each other, due to modulo-2 sum of parallelogram columns.
2. Consider properties of the actual polynomial divisor to obtain final coverage. □

Given x random errors, the probability that these errors are covered is $1-P_x$,

$$P_x = \frac{1}{C_{n\times s,x}} \times \sum_{j=0}^{x} E_x^{x-j} \times U_j \tag{4-5}$$

Notation

$C_{ns,x}$ number of combinations of x elements in a population of ns bits; the ways in which x erroneous bits can appear in the parallelogram
E_x^{x-j} number of configurations of x erroneous bits that map onto an equivalent serial polynomial in such a way that $x-j$ of these bits mask each other, while the remaining j bits appear as random erroneous bits
U_j parameter representing the fraction of undetectable equivalent serial input configurations among the ones affected by j random errors.

Note that:

1. $U_0 = 1$, because all erroneous bits mask each other
2. $E_x^{x-j} = 0$ whenever $x-j$ is an odd number, because modulo-2 sum cannot mask an odd number of changed bits;
3. U_j with $j \ne 0$ depends upon the characteristics of the divisor polynomial.

To evaluate precisely the detectability of random errors, it is necessary to refer to a given divisor polynomial, in order to compute the parameters U_j. An estimation independent from the divisor polynomial can be obtained by assuming a mean

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value for U_j, viz, $U_j = 2^{-s}$ for $j = 0, ..., x$, thus making no hypotheses on the specific characteristics of the polynomial itself. Since —

$$\sum_{j=0}^{x} E_x^{x-j} = C_{n \times s, x} \tag{4-6}$$

the resulting probability of not detecting random errors is again 2^{-s}. (Failures in the PSA itself can be seen as errors of classes 1-4.) □

In conclusion, the simple 2^{-s} is a reasonable evaluation of the probability of not detecting any type of error distribution — without requiring details either on the structure of the microprogram (therefore on the length of recovery segments) or on the characteristics of the divisor polynomial. As anticipated, the detection probability increases with s; on the other hand, a longer signature field implies a corresponding increase of the dimensions of the ROM (and, secondarily, of the PSA).

5. EFFECTIVENESS OF THE METHOD

To quantify the effective improvement introduced by our method as far as microprogram sequencing is concerned, it is necessary to weigh the error coverage (see section 4) with the increased probability of error occurrences due to the augmented silicon area that is sensitive to transient faults. To this purpose, we first evaluate the ratio of the silicon area of the nominal device *vs* the redundant area required to perform check and recovery actions.

5.1 *Area evaluations*

Functional elements constituting the augmented control unit — distinguished between nominal elements and redundant elements to achieve fault tolerance — are listed here.

Nominal elements

- Microprogram store ROM; the associated area is A_{ROM}.
- Microprogram sequencing logic (comprising microsubroutine stack, etc.); the associated area is A_{MSL}.
- Operation-code decoder and next-address generator; in many instances, both sub-units are implemented by a single PLA, with area A_{PLA}.
- Condition-code related logic, with area A_{COND}.
- Working registers; the associated area is A_{WREG}.
- Working register selection logic, characterized by area A_{RSEL}.

Redundant elements

- Extension of the micro-instruction store due to the s-bit long signature field and the control bit for check/recovery points. The related area is:

$$A_{SIG} = \frac{s+1}{m} \times A_{ROM} \tag{5-1}$$

Notation

m nominal length of a micro-instruction in the basic ROM.

- Extension of the microprogram due to insertion of dummy micro-instructions. Given the ratio R_D of dummy micro-instructions to nominal ones, the area increment is:

$$A_{DUM} = R_D \times \left(1 + \frac{s+1}{m}\right) \times A_{ROM} \tag{5-2}$$

- Combinational logic associated with the PSA. The related area A_{CPSA} derives partly from logic associated with condition-code treatment (which is independent of the PSA length) and partly from the PSA check and functional logic (linearly depending from the length of the PSA). In order to avoid that faults already identified by the PSA will pass undetected, the check upon the PSA status has to be guaranteed as fail-secure. For this reason, a self-checking checker is considered.
- Memory elements in the PSA; the related area, depending on the length s of the signature fields, is A_{MPSA}.
- Recovery registers; they constitute a critical section, since their failure leads to non recoverable error propagation. To grant the survival of the saved roll-back status in presence of faults affecting recovery registers, we adopt TMR and voting; see the classical scheme in [1]. The relative area is A_{RREG}.
- Logic driving the recovery phase; the related area is A_{REC}.

The areas of the nominal functional units and of the redundancy location are:

$$A_N = A_{ROM} + A_{MSL} + A_{PLA} + A_{COND} + A_{WREG} + A_{RSEL} \tag{5-3}$$

$$A_R = A_{SIG} + A_{DUM} + A_{CPSA} + A_{MPSA} + A_{RREG} + A_{REC} \tag{5-4}$$

Areas are evaluated by considering three classes of control units, ranging from relatively long microprograms of short, coded micro-instructions ($8K \times 32$-bit microwords: $m = 32$) to compact microprograms of long micro-instructions (256×128-bit microwords: $m = 128$). For each class, two implementations are envisioned, characterized by a working register bank containing — respectively — 16 16-bit and 16 32-bit registers (all provided with recovery registers), that represent average solutions in CPU design.

Moreover, considerations found in [8, 12] lead us to assume that a reasonable value for the silicon area A_I required by on-chip interconnections with respect to the total functional area is:

$$\frac{A_I}{A_N + A_R} = 0.1 \tag{5-5}$$

and a value for the ratio of dummy micro-instructions *vs* nominal

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ones, is $R_D = 0.05$ [12]. Chip area is expressed in terms of equivalent gates (area of a standard 2-input NAND), referring to a macrocell-based implementation of the device, using the macrocells in [20]. Results of these area evaluations are in table 1, while figure 6 plots the ratio of A_R to $A_N + A_I$ as a function of s for the classes of control units.

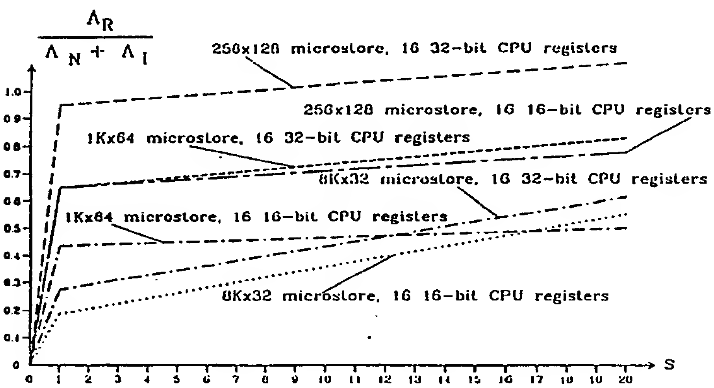

Figure 6. Ratio of Redundant-Area *vs* Length-of-Signature-Fields

5.2 *Safeness against transient faults*

We define the safeness (SAF) of the microprogrammed structure as the probability of obtaining correct sequencing operations. In a non-redundant structure, the safeness is obviously expressed by the probability of not having sequencing errors:

$$\mathrm{SAF}(A_{\mathrm{seq}}) = 1 - \Pr\{A_{\mathrm{seq}}\} \tag{5-6}$$

Notation

$\Pr\{A_{\mathrm{seq}}\}$ probability of occurrence of a sequencing error due to a transient fault affecting the silicon area dedicated to sequencing operations.

In the redundant structure foreseen by our method, the safeness becomes:

$$\mathrm{SAF}(A_{\mathrm{seq}}) = 1 - 2^{-s} \times \Pr\{A_{\mathrm{seq}}\} \tag{5-7}$$

since in this case each sequencing error has a probability 2^{-s} of passing undetected.

The total sequencing-sensitive area A_{seq} can be expressed as the sum of two terms characterized by different failure rates:

$$A_{\mathrm{seq}} = A_{\mathrm{seq}}^{c} + A_{\mathrm{seq}}^{m} \tag{5-8}$$

Notation

A_{seq}^{c} includes all combinational elements (i.e., elements in which a transient fault generates an error only if the element is being used when the fault occurs; note that this is also the case of faults affecting the memory access circuitries during read phases)

A_{seq}^{m} includes all memory elements (elements in which a transient fault — typically occurring during a memory write cycle — is "made static" until the next write operation takes place on the same memory location.

Referring to the functional elements listed in section 5.1, it results that

$$A_{\mathrm{seq}}^{c} = A_{\mathrm{PLA}} + A_{\mathrm{COND}} + A_{\mathrm{SIG}} + \frac{s+1}{m} \times A_{\mathrm{DUM}} + A_{\mathrm{CPSA}} + A_{\mathrm{REC}} \tag{5-9a}$$

$$A_{\mathrm{seq}}^{m} = A_{\mathrm{MSL}} + A_{\mathrm{MPSA}} \tag{5-9b}$$

Table 2 reports results of the evaluation of these areas, for the same control units of table 1.

To obtain the probability $\Pr\{A_{\mathrm{seq}}\}$ occurrence of a sequencing error, the following assumptions are now made:

TABLE 1

Silicon areas	8K×32		1K×64		256×128	
(equivalent gates)	16 reg.	32 reg.	16 reg.	32 reg.	16 reg.	32 reg.
$A_N + A_I$	50700	52600	14200	15800	8200	10000
A_R	5500+960s	7200+960s	2530+132s	4320+132s	2200+45s	3930+45s

TABLE 2

Silicon areas	8K×32		1K×64		256×128	
(equivalent gates)	16 reg.	32 reg.	16 reg.	32 reg.	16 reg.	32 reg.
A_{seq}^{c}	26680+957s	26680+957s	7273+127s	7273+127s	3531+40s	3531+40s
A_{seq}^{m}	2882+6s	2882+6s	715+6s	715+6s	363+6s	363+6s

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1. Transient faults are mutually statistically independent; the classical Poisson distribution can be accepted, expressed as $\text{poim}(\delta;\epsilon A)$.

Notation

δ number of transient faults

ϵ fault rate per area unit (per equivalent gate) and per micro-instruction execution time

2. Given the previous assumptions, transient faults appearing in the area A_{seq}^c reserved for combinational logic generate a lower fraction of actual errors than the ones appearing in the area A_{seq}^m occupied by memory devices. For these reasons, we can describe the error distribution again by a Poisson expression with two error rates: ϵ^c and ϵ^m, with $\epsilon^c < \epsilon^m$. The overall error rate per micro-instruction execution time is then $\epsilon^c A_{\text{seq}}^c + \epsilon^m A_{\text{seq}}^m$. As a consequence —

$$\Pr\{A_{\text{seq}}\} = 1 - \exp(-\epsilon^c A_{\text{seq}}^c - \epsilon^m A_{\text{seq}}^m) \tag{5-10}$$

Since $A_{\text{seq}} = A_{\text{seq}}^c + A_{\text{seq}}^m$ is related to A_R (and then to s), it is possible to plot the function SAF(s) as in figure 7. SAF(s) depends upon 4 parameters:

- ratio R_D of dummy micro-instructions to nominal ones
- average number n of micro-instructions in each microprogram segment (here assumed as 10)
- error rates $\epsilon^c = 9.26 \times 10^{-21}$ and $\epsilon^m = 9.26 \times 10^{-20}$ errors per equivalent gate per 100 nanoseconds [21].

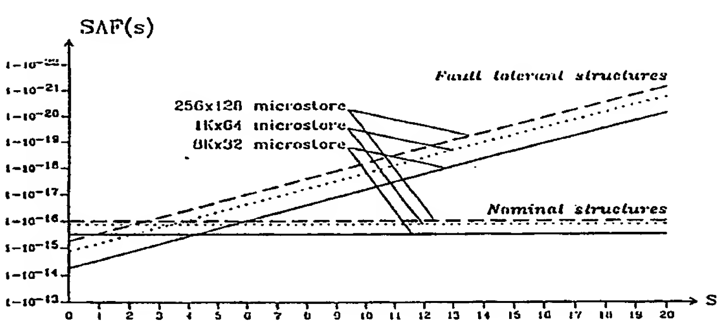

Figure 7. Safeness Against Transient Faults Affecting Sequencing

6. IMPACT OF THE TECHNIQUE ON YIELD & RELIABILITY

Our technique aims only at detecting and recovering from transient faults but, since it is envisioned for the design of VLSI devices, we must take into account the area increase not only as an immediate cost factor, but also for its effect upon yield and reliability. An area increase affects adversely both yield and reliability, unless redundancies are introduced specifically for overcoming production defects and run-time permanent faults [1, 8].

For yield evaluation, we follow the approach in [1, 8]. Given D (defect density per unit area) and A (total device area), the probability of obtaining a working device is:

$$p = \exp(-DA) \tag{6-1}$$

Considering that D is a stochastic variable and adopting for it the usual pdf [1], the yield is:

$$Y = [1 - \exp(-D_0 A)]/D_0 A \tag{6-2}$$

Notation

D_0 mean of the Cdf of D

Figure 8 plots the yield, $Y(s)$, for $D_0 = 5$ defects/cm^2 derived from [22] (corresponds to 7.06×10^{-5} defects per equivalent gate [20, 23]).

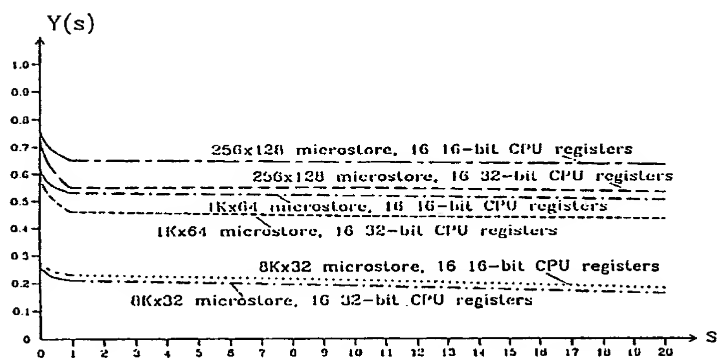

Figure 8. Yield *vs* Length-of-Signature-Fields

Consider now the reliability. Again, we assume the distribution of permanent failures to be Poisson [1]; this gives the well-known exponential curve for the reliability function *vs* time:

$$R(t) = \exp(-\lambda t) \tag{6-3}$$

Notation

λ failure rate (depends on the area).

This dependency is plotted in figure 9 as a function of the length of the signature field — extrapolating data in [1].

Having quantified both safeness against transient faults and reliability against permanent faults, it is now possible to evaluate the credibility of the device, defined as the probability of correct behavior: CR(s,t) = SAF(s)$R(t)$. In figure 10 the CR(s,t) has been plotted as a function of t, in the hypothesis of a signature field length equal to the number of bits of the microaddresses (required by the different classes of control units) + 1 (for 2-way conditional jumps, as shown in section 3).

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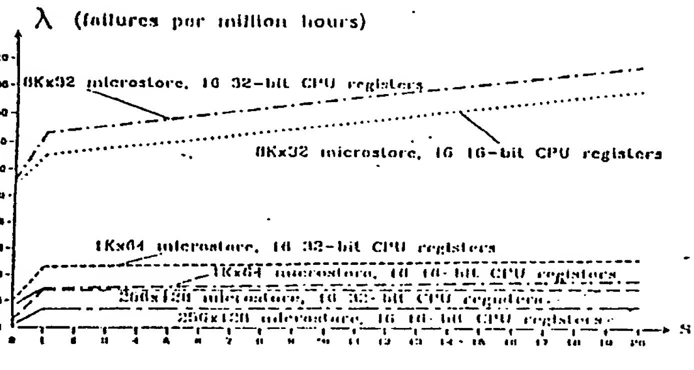

Figure 9. Permanent-Failure-Rate *vs* Length-of-Signature-Fields

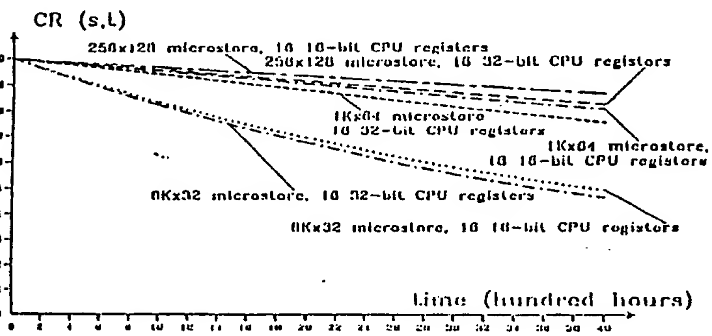

Figure 10. Credibility (s = microaddress length + 1)

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1/1 - (C) WPI / DERWENT
AN - 1977-42665Y ç24!
PR - JP19720117504 19721122
TI - Meat-like food prodn. from vegetable protein - by mixing porous tissue material from vegetable protein with thermal coagulant, pressing and heating
IW - MEAT FOOD PRODUCE VEGETABLE PROTEIN MIX POROUS TISSUE MATERIAL VEGETABLE PROTEIN THERMAL COAGULATE PRESS HEAT
PA - (FUKO) FUJI OIL CO LTD
PN - JP52018775B B 19770524 DW197724 000pp
ORD - 1977-05-24
IC - A23J3/00
FS - CPI
DC - D12 D13
AB - J77018775 Method comprises mixing a porous tissue material, produced from a vegetable protein (e.g. modified defatted soyabean protein) with a thermal coagulating material (e.g a mixt. of pepper and egg white) and the resulting mixt. is pressed and heated.
- Prod. has taste similar to that of whale meat.

PATENT COOPERATION TREATY

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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference 39887/NT	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
International application No. PCT/GB99/02111	International filing date *(day/month/year)* 02/07/1999	Priority date *(day/month/year)* 02/07/1998
International Patent Classification (IPC) or national classification and IPC A23J3/12		
Applicant MARS UK LIMITED et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 4 sheets, including this cover sheet.

   ☐ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consist of a total of sheets.

3. This report contains indications relating to the following items:

   - I ☒ Basis of the report
   - II ☐ Priority
   - III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   - IV ☐ Lack of unity of invention
   - V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations suporting such statement
   - VI ☐ Certain documents cited
   - VII ☐ Certain defects in the international application
   - VIII ☒ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
02/02/2000	05.10.2000
Name and mailing address of the international preliminary examining authority: European Patent Office - P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Tel. +31 70 340 - 2040 Tx: 31 651 epo nl Fax: +31 70 340 - 3016	Authorized officer Ainscow, J Telephone No. +31 70 340 3886

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB99/02111

I. Basis f th r p rt

1. This report has been drawn on the basis of (*substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments.*):

Description, pages:

1-7 as originally filed

Claims, No.:

1-15 as originally filed

Drawings, sheets:

1/3-3/3 as originally filed

2. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:
- ☐ the drawings, sheets:

3. ☐ This report has been established as if (some of) the amendments had not been made, since they have b n considered to go beyond the disclosure as filed (Rule 70.2(c)):

4. Additional observations, if necessary:

V. R asoned stat m nt under Articl 35(2) with r gard t n v lty, inventive step r industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	1-11
	No:	Claims	12-15
Inventive step (IS)	Yes:	Claims	1-11
	No:	Claims	12-15
Industrial applicability (IA)	Yes:	Claims	1-15
	No:	Claims	

2. Citations and explanations

see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

see separate sheet

Item V:

Reference is made to the following documents:

D1 = Van den Oord A.H.A. and J.J. Wesdorp, 1979

D2 = WPI/Derwent Abstract AN 86-303998 (SU-A-1220611)

D3 = WPI/Derwent Abstract AN 1977-42665Y (JP-B-52018775)

D4 = FR-A-2 315 858

D5 = WO-A-97-02760

D6 = DD-A-142 144

(D2 was not cited in the International Search Report)

1. A combined heat and hydrogen peroxide treatment of blood protein products such as "centriblood" was known in the art, see D1 and D2. It was also known in the art of texturising proteins that in order to obtain meat-like fibres, techniques like extrusion, expansion and spinning can be applied. D3 and D4 refer to coagulation and pressing of a soy protein material to obtain a meat-like product, but the structure is not specifically mentioned. D5 discloses the production of chunks having a striated appearance, by emulsification in an emulsion mill and coagulation.

However, the prior art does not suggest adding transition metal ions and an oxidising agent and compressing. Furthermore, the formation of a laminar structure by simple compressing could not have been foreseen on the basis of the cited prior art; D1 only mentions the coagulation into small beads.

Thus, the subject-matter of claims 1-11 is considered to be novel and to involve an inventive step (Articles 33(2) and (3) PCT).

2. The subject-matter of claims 12 and 13 is not new in view of D5 and D6, which disclose the preparation of a protein chunk, wherein a protein solution, containing blood, is heat-coagulated and stirred intensively.

3. Claims 14 and 15 cannot be considered new or inventive as they are not clear (see below).

Item VIII:

Claims 14 and 15 are not clear as they refer to the description Article 6, Rule 6.2(a) PCT in that the matter for which protection is sought is not clearly defined. The same applies to the expression "as herein defined" in claim 6.

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(54) Title: COAGULATED PROTEIN

(57) Abstract

The invention provides a method of manufacturing a coagulated protein chunk. The method comprises adding transition metal ions and an oxidising agent to a protein, compressing the resulting reaction product to form a chunk having a laminar structure. An edible chunk comprising a major proportion of protein and having a fibrous laminar internal structure is also described.

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COAGULATED PROTEIN

The present invention relates to the preparation of a chunk of coagulated protein, and to the coagulated protein chunk itself.

Conventionally, protein can be coagulated in a variety of ways, for example by heating it or treating it with acid. It has now been found that a protein may be coagulated by adding transition metals ions and an oxidising agent to a protein and compressing the reaction product of the transition metal ions, the oxidising agent and the protein. A textured solid mass is produced which may have an internal texture similar to that of cooked meat.

According to the invention there is provided a method of forming coagulated protein chunk comprising adding transition metal ions and an oxidising agent to a protein and compressing the resulting reaction product to form a chunk having a laminar structure.

It is believed that the transition metal ions and the oxidising agent react to form free radicals and that the free radicals then react with the protein to coagulate it.

According to the invention there is also provided a method of forming a coagulated protein chunk which comprises: generating free radicals by reacting transition metal ions with an oxidising agent; reacting the free radicals with a protein; and compressing the reaction product of the free

radicals and the protein to form a chunk having a laminar structure.

The reaction of transition metal ions with the oxidising agent and/or the reaction of free radicals with the protein may be heated.

Preferably the oxidising agent is present at least 0.5% by weight of the protein.

Preferably the transition metal ions are present at least 0.5% by weight of the protein.

Preferably the protein comprises at least about 5%, preferably at least about 10% by weight of the reaction mixture.

Preferably the transition metal ions are ferrous ions.

Preferably the oxidising agent is hydrogen peroxide.

In a preferred embodiment of the invention there is provided a method of forming a blood chunk comprising heating a blood fraction (as defined below); treating the heated blood fraction with hydrogen peroxide; and compressing the reaction product of the blood fraction and hydrogen peroxide. The blood fraction is defined herein as comprising from about 14% to about 40% protein and about 35% to 45% red blood cells. The blood fraction may be formed in any way. The blood fraction may be the hæmoglobin fraction of blood (as defined below). Alternatively, the blood fraction may be formed by removing water from whole blood to concentrate it so that it comprises from about 14% to about

40% protein and about 35% to 45% red blood cells. The blood fraction may be reconstituted from purified protein and red blood cells. By the haemoglobin fraction is meant the residue from whole blood once the plasma, or most of the plasma, has been removed. The haemoglobin fraction consists of red and white blood cells with a residue of plasma. The haemoglobin fraction typically contains from about 14% to about 40% protein and about 35% to about 45% red blood cells. The remainder is mainly water together with other blood components.

It will be appreciated that the blood fraction is a source of protein and ferrous ions. When a blood chunk is formed according to this preferred embodiment of the invention, no addition of transition metal ions is required for coagulation of the protein. When other sources of protein are used, it may be desirable to add additional transition metal ions.

Preferably the hydrogen peroxide is added to the blood fraction at at least 0.5% by weight. There does not appear to be a significant upper limit to the concentration of hydrogen peroxide in the reaction mixture which is effective to cause the desired reaction to take place; concentrations of up to 3% (by weight) have been found satisfactory.

Preferably, compression is carried out at a temperature greater than 60°C.

Preferably the blood fraction is heated to between 60°C and 80°C before addition of the hydrogen peroxide.

Preferably the blood fraction comprises at least about 10%, more preferably at least about 15%, by weight protein. At lower protein concentrations, the reaction product does not absorb all the water present in the reaction mixture. Such products are useful and their manufacture falls within the scope of the present invention; however, it will usually be necessary to remove the proteinaceous material from the unabsorbed water before it is used.

Additives may be included in the blood fraction to modify the nutritional content and flavour of the chunks. It is preferred that the pH of blood fraction is no less than 4, and that it is no greater than 9.

Compression of the reaction product of the blood fraction and hydrogen peroxide can be carried out on the reaction product as it is formed, or the reaction product can be stored and then subjected to heating, for example by microwave radiation, prior to compressing. Alternatively, the reaction product may be steamed to give a product having a jelly-like texture. The steaming can be carried out with meat juices or other flavoured aqueous media to impart particular flavours to the product.

The product can be dried, preferably at about 60°C, to produce hard, crunchy chunks, which are useful as a dry pet food.

The reaction product of the blood fraction and hydrogen peroxide can be compressed under its own weight.

The reaction product may be compressed as a result of restriction of any expansion of the reaction product caused

by evolution of gas as the transition metal, oxidising agent and protein react.

The pressure at which the reaction product of the blood fraction and the hydrogen peroxide is compressed to achieve the laminar internal structure is not critical; a pressure of up to about 400 kPa is preferred.

Also according to the invention there is provided an edible chunk comprising a major proportion of protein, preferably blood protein, and having a fibrous, laminar internal structure.

The invention will be further described, by way of example, with reference to the drawings in which;

Figure 1 shows schematically a method according to a first embodiment of the invention;

Figure 2 shows schematically a method according to a second embodiment of the invention; and

Figure 3 shows schematically a method according to a third embodiment of the invention.

The methods according to the invention shown in the drawings include the following common features. The hæmoglobin fraction of blood is pumped from a tank 10 by a peristaltic pump 12 to a steam infuser 14 where the hæmoglobin is heated to about 75°C. The heated hæmoglobin passes from the steam infuser 14 to a high shear mixer reactor 16, such as a Dispax reactor. In the Dispax reactor, the hæmoglobin is reacted with hydrogen peroxide pumped from a hydrogen

peroxide tank 18 by a hydrogen peroxide pump 20. In the reactor 16, the hæmoglobin and the hydrogen peroxide are mixed efficiently. Preferably, the reactor is a high shear, low volume mixer to ensure adequate mixing of the two components.

In the first embodiment of the invention, shown in Figure 1, the foam reaction product 22 is deposited in a tray 24. The reaction product 22 can be allowed to be compressed by its own weight, in which case the solid mass produced is elastic and can be cut up to provide elastic chunks. Alternatively, pressure can be applied to the reaction product 22 in the tray by application of a pressure plate 26. On release of the pressure plate a solid product 28 having a fibrous, laminar internal structure is produced, which can then be cut into chunks 30 as at 32.

In the second embodiment of the invention, shown in Figure 2, the reaction product 22 from the reactor 16 is passed to a piston pump 40 in which the reaction product is compressed. As the reaction product 22 leaves the piston pump 40, it is diced as at 42 to produce chunks 44 having a fibrous, laminar internal structure.

In the third embodiment of the invention, shown in Figure 3, the reaction product 22 leaves the reactor 16 through a disperser 50, from where it passes into a mouth formed by the widely separated ends of two converging continuous belts 52, 44. The reaction product is compressed between the two continuous belts, and the resulting solid sheet 56 is cut into chunks 58 as it leaves the continuous belts 52, 54, as at 60. Again, the chunks produced have a fibrous, laminar internal structure.

The chunks have a fibrous, laminar internal structure, similar to that of meat chunks, so that the chunks can be readily used in canned food stuffs such as pet foods to provide a protein source which is analogous in appearance and texture to meat.

CLAIMS

1. A method of forming a coagulated protein chunk comprising adding transition metal ions and an oxidising agent to a protein and compressing the resulting reaction product to form a chunk having a laminar structure.

2. A method of forming a coagulated protein chunk comprising: generating free radicals by reacting transition metal ions with an oxidising agent; reacting the free radicals with a protein; and compressing the reaction product of the free radicals and the protein.

3. A method according to claim 1 or 2 in which the compression is carried out at a temperature greater than 60°C.

4. A method according to any preceding claim in which the compressed product is dried.

5. A method according to any preceding claim further comprising steaming the reaction product of the transition metal ions, the oxidising agent and the protein.

6. A method according to any preceding claim in which a blood fraction (as herein defined) is reacted with transition metal ions and the oxidising agent.

7. A method according to any preceding claim in which the transition metal ions are ferrous ions.

8. A method according to any preceding claim in which the oxidising agent is hydrogen peroxide.

9. A method according to any preceding claim in which the oxidising agent is present at at least 0.5% by weight of the protein.

10. A method according to any preceding claim in which the transition metal ions are present at at least 0.5% by weight of the protein.

11. A method according to any preceding claim in which the protein comprises at least about 5%, preferably at least about 10%, protein by weight of the reaction mixture.

12. Coagulated protein formed by a method according to any of claims 1 to 11.

13. An edible chunk comprising a major proportion of protein, preferably blood protein, and having a fibrous, laminar internal structure.

14. A method substantially as described.

15. A chunk substantially as described.

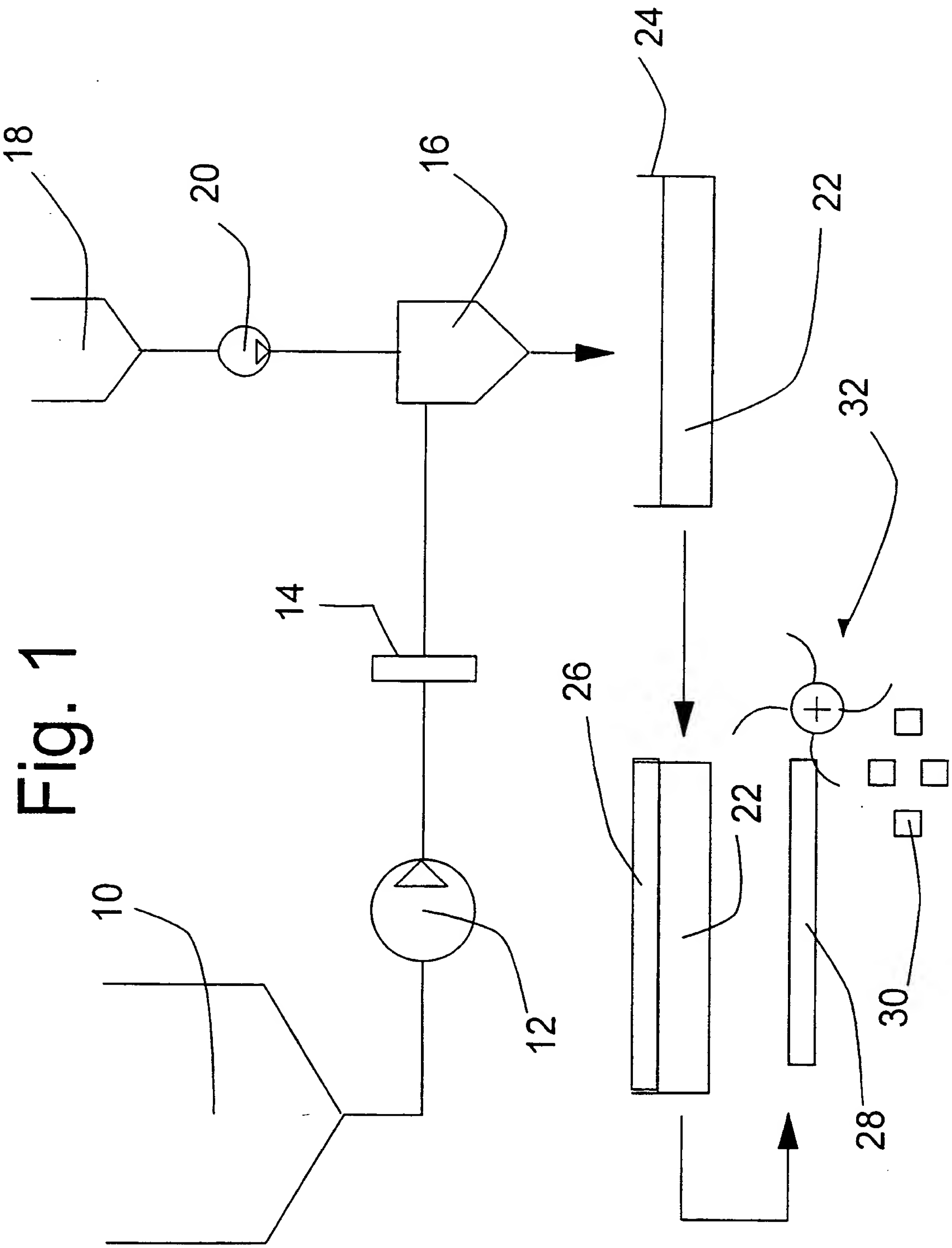

Fig. 1

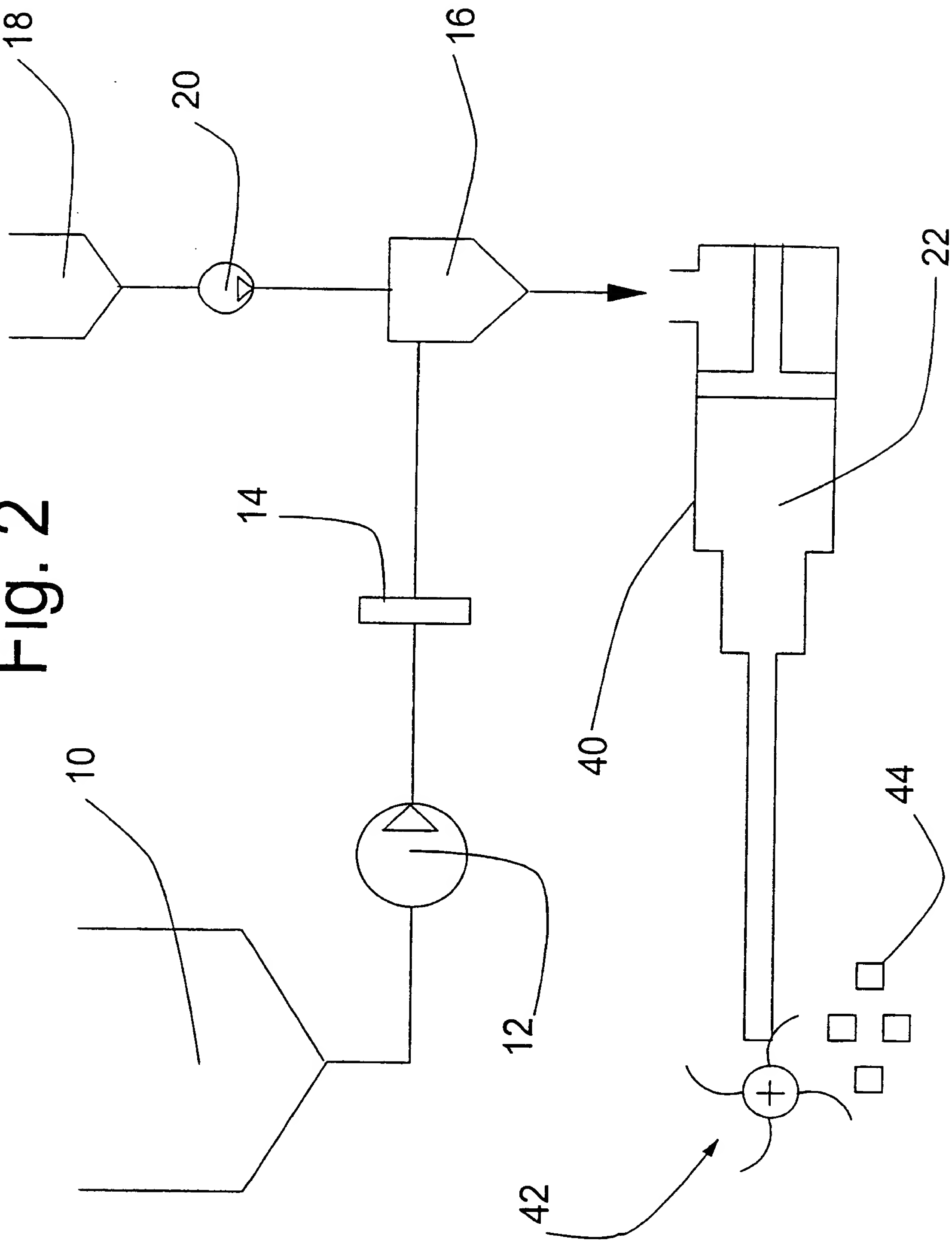

Fig. 2

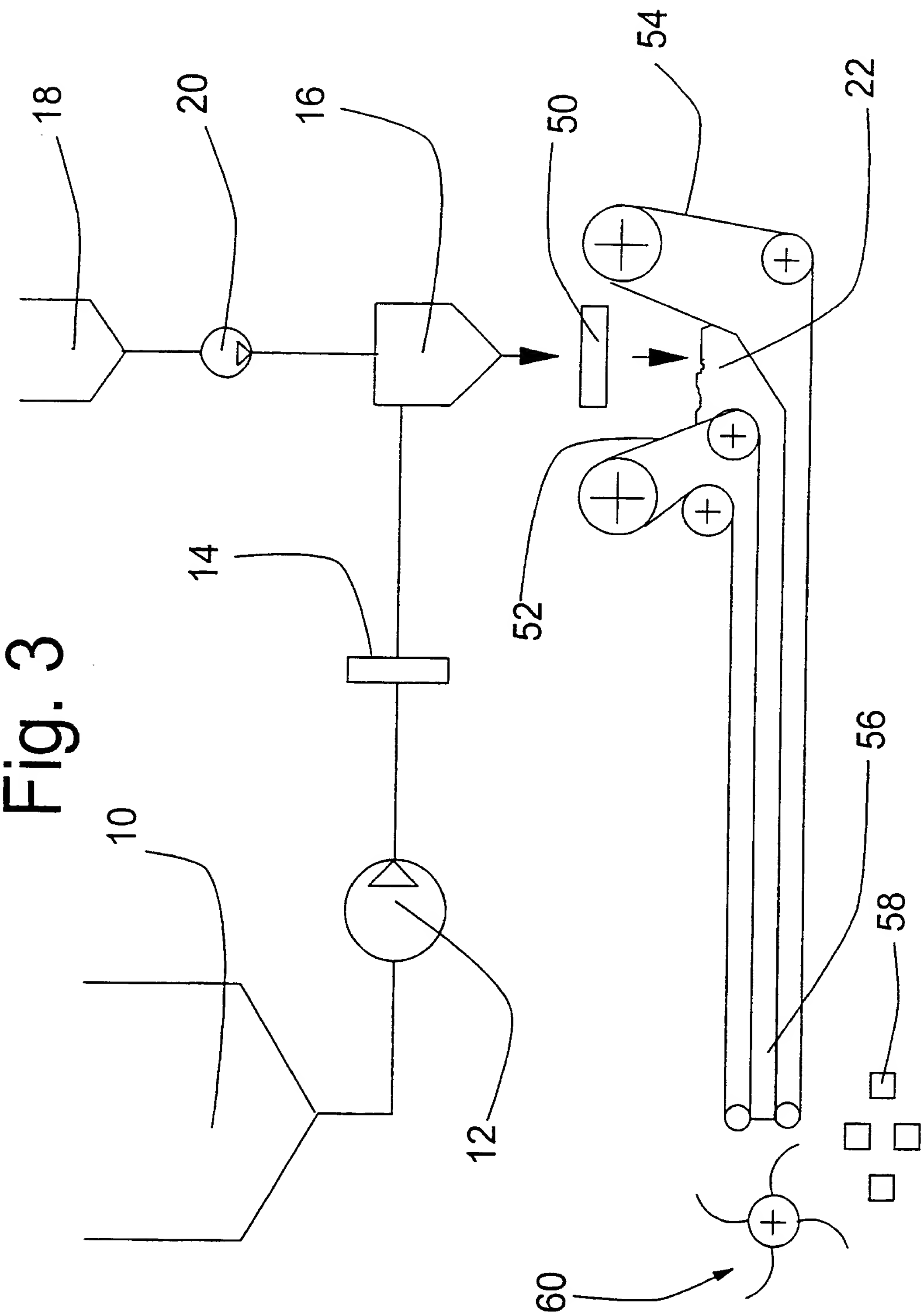

Fig. 3

INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 99/02111

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 A23J3/12 A23K1/04 A23J3/22 A23J3/28

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 A23J A23K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	OORD A H A VAN DEN ET AL: "Decolouration of slaughterhouse blood by treatment with hydrogen peroxide" ABSTRACTS AND COMMUNICATIONS. EUROPEAN MEETING OF MEAT RESEARCH WORKERS, 1 January 1979 (1979-01-01), page 827/828 XP002081374 the whole document ---	1-11
A	ANTOLA A ET AL: "TOLERANCE TO TRANSIENT FAULTS IN MICROPROGRAMMED CONTROL UNITS" IEEE TRANSACTIONS ON RELIABILITY, vol. 39, no. 5, 1 December 1990 (1990-12-01), pages 535-546, XP000208707 ISSN: 0018-9529 the whole document ---	1-11
	-/--	

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier document but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
25 October 1999	26/11/1999

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	De Jong, E

1

Form PCT/ISA/210 (second sheet) (July 1992)

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication,where appropriate, of the relevant passages	Relevant to claim No.
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A	the whole document ---	1-11
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/GB 99/02111

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AMT FÜR ERFINDUNGS- UND PATENTWESEN

PATENTSCHRIFT 142144

Wirtschaftspatent
Erteilt gemäß § 5 Absatz 1 des Änderungsgesetzes zum Patentgesetz

In der vom Anmelder eingereichten Fassung veröffentlicht

Int. Cl.3

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(71) siehe (72)

(72) Kummer, Siegfried, Dipl.-Ing., DD; Raeuber, Hans-Jörg, Prof. Dr.sc.techn., DD; Lchagwaa, Gurragtschagijn, Dipl.-Ing., MN

(73) siehe (72)

(74) Technische Universität Dresden, Direktorat für Forschung, BfSN, 8027 Dresden, Mommsenstraße 13

(54) Verfahren zur Herstellung von fibrillären und lamellaren Eiweißstrukturen

(57) Die Erfindung betrifft ein Verfahren zur Herstellung von fibrillären und lamellaren Eiweißstrukturen aus pflanzlichen oder tierischen Eiweißlösungen, z.B. aus Magermilch, Molke oder einem Gemisch von beiden, für die Produktion neuer Eiweißnahrungsmittel. Die Erfindung hat das Ziel, ein weiteres Verfahren zur Herstellung texturierter Eiweiße aus pflanzlichen und tierischen Eiweißlösungen anzugeben. Die Erfindung löst die Aufgabe, aus pflanzlichen und tierischen Eiweißlösungen fibrilläre und lamellare Eiweißstrukturen herzustellen, und dabei die Einfachheit des Rührverfahrens in etwa mit den Qualitätsvorzügen von gesponnenen Eiweißfasern zu verbinden. Erfindungsgemäß wird die Aufgabe dadurch gelöst, daß den Eiweißlösungen 5 bis 33% Blutplasma zugesetzt, das Gemisch bei einer Temperatur unterhalb der Denaturierungsgrenze, vorzugsweise 1 bis 2 °C darunter, 10 bis 60 min einer Ruhephase unterworfen, bei 80 bis 100 °C thermisch nachbehandelt und gegebenenfalls zur Erzielung fibrillärer Eiweißstrukturen während der thermischen Nachbehandlung intensiv zerrührt wird.

4 Seiten

Verfahren zur Herstellung von fibrillären und lamellaren Eiweißstrukturen

Anwendungsgebiet der Erfindung

Die Erfindung betrifft ein Verfahren zur Herstellung von fibrillären und lamellaren Eiweißstrukturen aus pflanzlichen oder tierischen Eiweißlösungen, z.B. aus Magermilch, Molke oder einem Gemisch von beiden, für die Produktion neuer Eiweißnahrungsmittel. Die Anwendung kann in Kombinationsfleischprodukten, Teilsimulaten und in gewissem Umfang als Vollsimulat oder als eigenständiges, neues Produkt erfolgen.

Charakteristik der bekannten technischen Lösungen

Zur Herstellung texturierter Eiweiße aus Eiweißlösungen sind verschiedene Verfahren bekannt.

Es werden Eiweißfasern gesponnen, Eiweißfäden extrudiert, Strukturen ausgefroren oder mittels Pilzorganismen schwammartige Strukturen gebildet und anschließend zu Texturen gewalzt.

Die schnelle Erhitzung koagulierbarer Eiweiße unter intensiver Rührwirkung führt ebenfalls zu fasrigen Strukturen. Im Gegensatz zum Spinnprozeß ist das Verfahren einfacher. Es entfallen verschiedene Verfahrensstufen, insbesondere die chemische Nachbehandlung und Neutralisation der Fasern. Andererseits ist die Qualität der durch Rühren erzielten Struktur nur minderwertig.

Es ist auch bekannt, durch den Zusatz von grenzflächenaktiven Substanzen zu Weizengluten eine fasrige Struktur zu bilden. Dieses Verfahren ist nur für den Einsatz des genannten Rohstoffes anwendbar.

Ziel der Erfindung

Die Erfindung hat das Ziel, ein weiteres Verfahren zur Herstellung texturierter Eiweiße aus pflanzlichen und tierischen Eiweißlösungen anzugeben.

Darlegung des Wesens der Erfindung

Die Erfindung löst die Aufgabe, aus pflanzlichen und tierischen Eiweißlösungen fibrilläre und lamellare Eiweißstrukturen herzustellen, und dabei die Einfachheit des Rührverfahrens in etwa mit den Qualitätsvorzügen von gesponnenen Eiweißfasern zu verbinden.

Erfindungsgemäß wird die Aufgabe dadurch gelöst, daß den Eiweißlösungen 5 bis 33 % Blutplasma zugesetzt, das Gemisch bei einer Temperatur unterhalb der Denaturierungsgrenze, vorzugsweise 1 bis 2 °C darunter, 10 bis 60 min. einer Ruhephase unterworfen, bei 80 bis 100 °C thermisch nachbehandelt und ggf., zur Erzielung fibrillärer Eiweißstrukturen, während der thermischen Nachbehandlung intensiv zerrührt wird. Das Gemisch aus Eiweißlösungen und Blutplasma bildet in der Ruhephase eine Koagulationsstruktur aus. In der anschließenden Behandlung erfolgt die Verfestigung des Gels zu einer Kondensationsstruktur. Es bilden sich lamellare Eiweißstrukturen. Wird während der thermischen Nachbehandlung intensiv zerrührt, bilden sich fibrilläre Eiweißstrukturen, die von der freigewordenen Flüssigkeit getrennt und mit einem Bindemittel zu einem Texturat verarbeitet werden können.

Ausführungsbeispiel

Ein Gemisch aus Magermilch, Molke und Blutplasma im Verhältnis 2 : 2 : 1 wird auf einen pH-Wert von 6,4 eingestellt und auf 45 °C erwärmt. Bei dieser Temperatur 15 min. gehalten, geliert das Gemisch. Danach erfolgt eine Erwärmung auf 95 °C bis das Eiweiß völlig denaturiert ist. Der so erhaltene Eiweißblock weist eine lamellare Struktur auf.

Erfindungsanspruch

1. Verfahren zur Herstellung von fibrillären und lamellaren Proteinstrukturen aus pflanzlichen oder tierischen Eiweißlösungen, z.B. aus Magermilch, Molke oder einem Gemisch von beiden, gekennzeichnet dadurch, daß den Eiweißlösungen 5 bis 33 % Blutplasma zugesetzt, das Gemisch bei einer Temperatur unterhalb der Denaturierungsgrenze, vorzugsweise 1 bis 2 °C darunter, 10 bis 60 min. einer Ruhephase unterworfen, bei 80 bis 100 °C thermisch nachbehandelt und ggf., zur Erzielung fibrillärer Eiweißstrukturen, während der thermischen Nachbehandlung intensiv zerrührt wird.

XP-002120043

P.D. 1994
p. / = 1

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AN  - 94-1-07-s0026 FSTA
DT  - J Journal
TFT - S - Meat, poultry & game
TI  - Extrusion technology of processing and utilization of slaughterhouse
      blood.
AU  - Fajvisevskij M L;  Lisina T N
PUB - Tehnologija Mesa
    - 1993
    - VNIIMP, Moscow, Russia
IRN - ISSN 0494-9846
VOL - 34
NR  - 2/3
PG  - 58 - 60
LA  - Sh, en
IW  - Cereal products;   Blood
    - Cereal products
AW  - BLOOD;   CEREAL PRODUCTS
    - ISBN 0-85954-329-3
AB  - A blood product made by extrusion processing of a mixture of blood,
      wheat flour, corn starch and dried milk is described. Data are given
      for mineral, vitamin, amino and fatty acid compositions of the
      extruded product, together with expansion characteristics and
      nutritional value (assessed in rat feeding trials). Results showed
      that the extruded product had high Fe availability. It is concluded
      that extrusion processing of blood/cereal mixtures is a
      useful method for manufacture of blood-containing foods for use as a
      dietary Fe source.
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XP-002120046

1/1 - (C) WPI / DERWENT
AN - 1980-61444C ç25!
PR - JP19790002411 19790112
TI - Dewatering organic sludge formed in sewage treatment - by adding iron salt and hydrogen peroxide, heating, opt. neutralising and filtering
IW - DEWATER ORGANIC SLUDGE FORMING SEWAGE TREAT ADD IRON SALT HYDROGEN PEROXIDE HEAT OPTION NEUTRALISE FILTER
PA - (TSUH) TSUKISHIMA KIKAI CO
PN - JP55094698 A 19800718 DW198035 000pp
ORD - 1980-07-18
IC - C02F11/14
FS - CPI
DC - D15
AB - J55094698 Organic sludge generated in treatment of sewage or industrial waste waters is dewatered effectively with improved filtration rate by adding iron salt, e.g. $FeSO_4$ and $FeCl_3$ etc. in amt. 5,000-15,000 ppm, then adding H_2O_2 in amt. 1,000-3,000ppm, and subsequently heating it at 60-100 degrees C for 10-60 mins. followed, if necessary, by pH adjustment until neutral with strong alkali, e.g. NaOH etc. to prevent corrosion of appts.
- H_2O_2 serves as strong oxidising agent esp. in the presence of Fe ion, and the molar ratio $FeSO_4/H_2O_2$ is pref. within the range of 0.8-1.2. The oxidising reaction is accelerated by heating, and proteins contained in the sludge are coagulated in the heat treatment; filterability and filtration rate are improved by heating, water content of dewatered filter cake is largely lowered by heating. Solid-liq. sepn. becomes easy after heat treatment, and content of filtrate is decreased after filtration of con. sludge, bad odour is not generated, filter assistant, e.g. $Ca(OH)_2$ is not necessary, and amt. of filter cake generated is very little.